

Preparation and anisotropic properties of textured structural ceramics: A review

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Abstract: Ceramics are usually composed of randomly oriented grains and intergranular phases, so their properties are the statistical average along each direction and show isotropy corresponding to the uniform microstructures. Some methods have been developed to achieve directional grain arrangement and preferred orientation growth during ceramic preparation, and then textured ceramics with anisotropic properties are obtained. Texture microstructures give particular properties to ceramics along specific directions, which can effectively expand their application fields. In this review, typical texturing techniques suitable for ceramic materials, such as hot working, magnetic alignment, and templated grain growth (TGG), are discussed. Several typical textured structural ceramics including α -Al₂O₃ and related nacre bioinspired ceramics, Si₃N₄ and SiAlON, h-BN, MB₂ matrix ultra-high temperature ceramics, MAX phases and their anisotropic properties are presented.

Keywords: texture; structural ceramics; anisotropic properties; strengthening and toughening mechanisms

1 Introduction

Since the periodicity and density of the atoms in single crystals are not identical along different directions, the physical and chemical properties of single crystals along different directions are various. But most materials, either metals or ceramics, are polycrystalline materials composed of unoriented grains, resulting in their isotropic

properties. When some external conditions, such as stress fields, electromagnetic fields, and temperature fields, are applied during the process of material preparation, grains can be preferentially aligned along specific crystallographic directions, forming texture microstructures [1–3]. Performances of materials along preferred crystal lattice orientation can be enhanced by texturing so that they can be applied to more harsh service environments [4–6].

For many metallic materials, their slip systems can be activated at suitable temperature, and then grains can be easily oriented by deformation, such as rolling

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and extrusion [7–12]. Subsequent heat treatment of deformed metal can result in oriented nucleation of recrystallization and preferred orientation growth [13–17]. By appropriate deformation and heat treatment, texture degree and grain size of metals can be adjusted, and the required anisotropic properties can be obtained [18–21]. Unlike metals, atoms in ceramics are mainly interconnected by covalent and ionic bonds, which are strong enough to prevent plastic deformation. So deformation techniques are not suitable for texture formation of ceramics. Specific techniques, such as hot working, magnetic alignment, and templated grain growth (TGG), have been invented, which can effectively promote the preferred orientation of ceramic grains [2,22,23].

Textured ceramics have many superior properties compared with ceramics composed of randomly oriented grains. As for functional ceramics, texture microstructures can increase the critical current density of superconductors [24–26], increase the electrical conductivity of ionic conductors [27,28], heighten the magnetic anisotropy of hexaferrite ceramics [29–31], and improve the electromechanical coupling coefficient of piezoelectric ceramics [32–35]. As for structural ceramics, fracture toughness along specific directions increases with the incorporation of oriented grains, which favor the mechanisms of crack deflection, crack bridging, and grain pull-out [36–39]. Figure 1 shows the schematic diagram of the microstructure of a lamellar textured porous α -alumina (α -Al₂O₃) specimen prepared by hot-pressing (HP) and its anisotropic mechanical properties and thermal conductivities [40]. Due to the texture microstructure, it possesses higher thermal conductivity

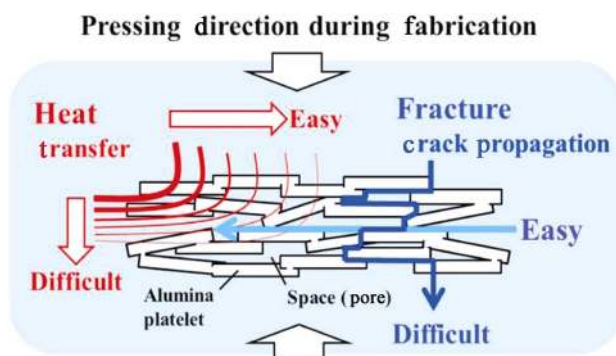


Fig. 1 Schematic diagram of the microstructure and anisotropic mechanical properties and thermal conductivities of a lamellar textured porous α -Al₂O₃ specimen prepared by HP. Reproduced with permission from Ref. [40], © Elsevier Ltd and Techna Group S.r.l. 2015.

along the lamella and better mechanical properties perpendicular to the lamella.

There are many examples of textured materials with excellent performances in natural creatures. Natural materials, such as nacre, bone, and bamboo, usually combine stiff and soft components and possess hierarchical structures [41]. For example, many skeletal tissues consist of organic fibrils and mineral particles [42,43]. In the exoskeleton of *Homarus americanus*, the organic matrix consists of α -chitin fibrils and some non-crystalline proteins. Most of the α -chitin lattice cells are oriented with the long crystallographic axis towards the surface of the exoskeleton to provide mechanical support for the body [44]. Another typical example is that many mollusks protect themselves from predators by their hard external shells consisting of a brittle external calcite layer and a tough internal nacre layer. The nacre layer is composed of aragonite, chitin, and proteins, and possesses the brick-and-mortar structure employing a variety of toughening mechanisms such as crack deflection and microbuckling to induce a gradual “graceful failure” [45]. Inspired by specific structures of natural creatures, much research has been put into textured materials possessing hierarchical structures [46–58].

There have already been some reviews about textured metals and functional ceramics [32,59–63]. But there are few summaries about textured structural ceramics. In this review, an overview of preparation methods of textured structural ceramics is given. In addition, the state of the art of several typical textured structural ceramics and their anisotropic properties are discussed. The aim of this review is to give a brief introduction of textured structural ceramics for new researchers in this field and to provide some useful references for them.

2 Characterization of texture

Texture can be divided into morphological texture where anisometric grains are oriented and crystallographic texture where grains have preferred crystal lattice orientation. These axis orientations (the c -axis orientation, the a,b -axis orientation, etc.) in the remainder of this review all refer to the crystallographic axes system. Morphological texture and crystallographic texture usually coexist for textured structural ceramics. Morphological texture can be characterized by microscopes such as optical microscope (OM), scanning electron microscope

(SEM), transmission electron microscope (TEM), etc. Crystallographic texture can be characterized by pole figures and inverse pole figures obtained with X-ray diffraction (XRD) and electron back scattering diffraction (EBSD) using SEM, Euler maps obtained with EBSD using SEM, etc. The diffraction intensities of special crystal planes obtained by XRD and neutron diffraction (ND) can also be used to characterize crystallographic texture. ND is very useful to analyze large bulk materials as neutrons have the highest penetration among neutrons, electrons, and X-ray [64], and it can also be used for *in situ* observation of texture development due to its low absorption [65–67]. As the characterization methods of texture are usually used in the following parts of this review, they are discussed here firstly.

2.1 Characterization of morphological texture

OM and SEM can provide visualized morphology information of textured materials. TEM can be used to observe morphological microstructure and determine grain orientation in a small area of several micrometers. Figure 2 shows the polished surface morphology of textured alumina observed by OM, indicating that plate-like alumina grains were oriented perpendicular to the magnetic field that applied during slip casting [68]. But in most cases, especially for the ceramics with fine grain sizes, SEM is used more frequently due to its higher resolution than OM. Figure 3 shows the fracture morphology of textured alumina ceramics [69]. Although the grain size is only about 1–5 μm, texture characteristics can be observed clearly. And the relationship between magnetic field and grain preferred orientation is the same as that in Fig. 2. Figure 4 shows the TEM images of textured h-BN-MAS composites prepared from h-BN powders with different sizes, where grain size and morphology are clearly seen [70]. The grain orientation is more obvious for textured specimen prepared using h-BN powders with large size.

For textured ceramics mainly containing elongated grains, orientation factor f_0 can be calculated from SEM morphology images to quantitatively characterize morphological texture degree, which is defined as Eq. (1) [71]:

$$f_0 = \frac{2}{N} \sum_{i=1}^N \cos^2 \theta_i - 1 \quad (1)$$

where N is the number of elongated grains, and θ_i is the tilt angle between the real grain orientation and the

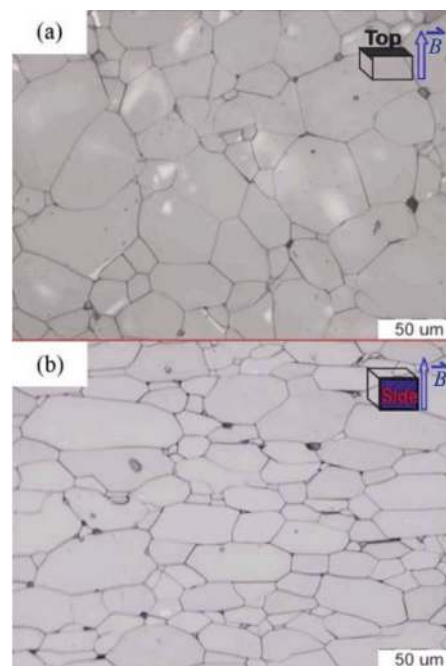


Fig. 2 OM thermal etched surfaces of textured alumina ceramics: (a) perpendicular to the magnetic field direction; (b) parallel to the magnetic field direction. Reproduced with permission from Ref. [68], © Elsevier Ltd and Techna Group S.r.l. 2012.

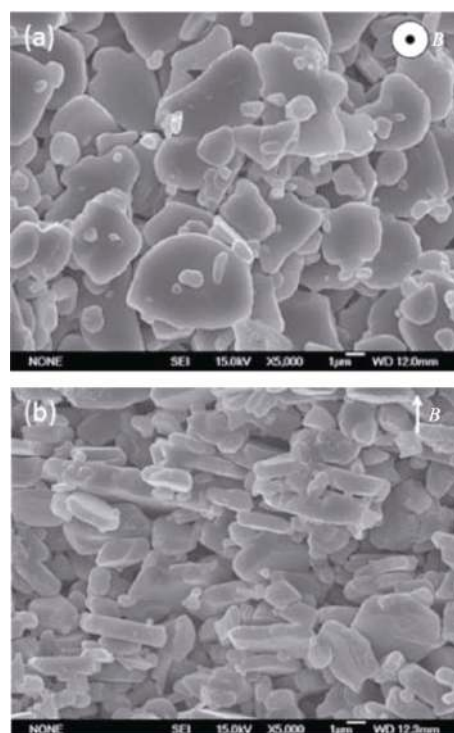


Fig. 3 SEM fracture morphologies of textured alumina ceramics: (a) perpendicular to the magnetic field direction; (b) parallel to the magnetic field direction. Reproduced with permission from Ref. [69], © WILEY - VCH Verlag GmbH & Co. KGaA, Weinheim 2015.

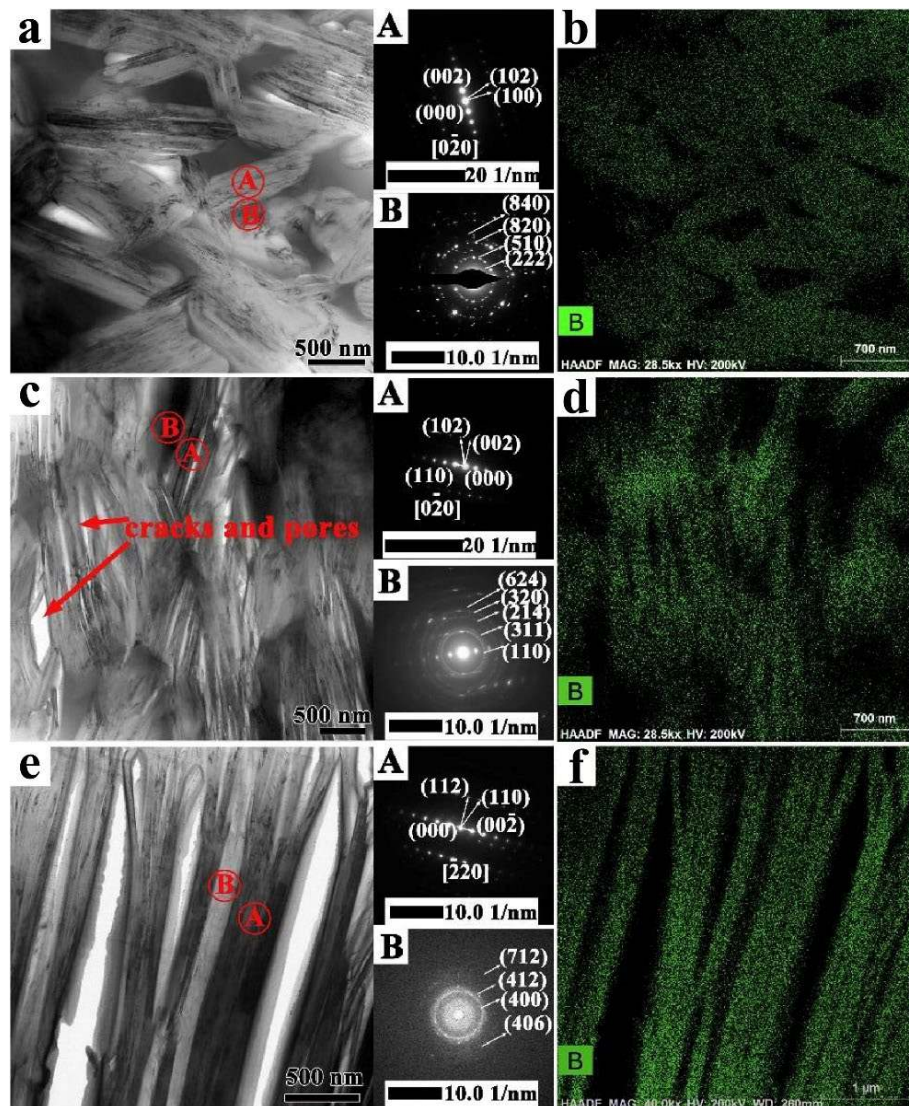


Fig. 4 Bright field TEM images, corresponding SAED patterns, and elemental distributions of boron for textured h-BN-MAS composites prepared from h-BN powders with different sizes: (a, b) 0.5 μm ; (c, d) 5.0 μm ; (e, f) 11.0 μm . Reproduced with permission from Ref. [70], © Elsevier Ltd. 2018.

ideal texture orientation. N and θ_i can be obtained by image analysis software. For a sample with randomly oriented grains, $f_0 = 0$. For samples with perfect texture parallel and perpendicular to the ideal texture orientation, $f_0 = 1$ and -1 , respectively.

2.2 Characterization of crystallographic texture

2.2.1 Pole figures, inverse pole figures, and Euler maps

EBSD device attached to SEM can provide Kikuchi patterns and further give orientation maps (Euler maps) [72–75]. The schematic diagram of EBSD is shown in Fig. 5, where the two diffraction cones (Kossel cones) of two sides of one crystal plane form the Kikuchi

band on the screen [76].

EBSD and XRD can both provide pole figures and inverse pole figures, which can provide the orientation information on one sample surface. Pole figures show the projection of the crystal orientation in the sample coordinate system, and inverse pole figures show the projection of the sample orientation in the crystal coordinate system [77]. To obtain the $\{hkl\}$ pole figure of one polycrystalline sample by XRD, the 2θ angle determined by Bragg's law should be fixed and the sample should be rotated in the coordinate system so that diffraction can occur at all $\{hkl\}$ crystal planes in every crystal [78]. EBSD can provide pole figures by indexing Kikuchi patterns. EBSD possesses a much

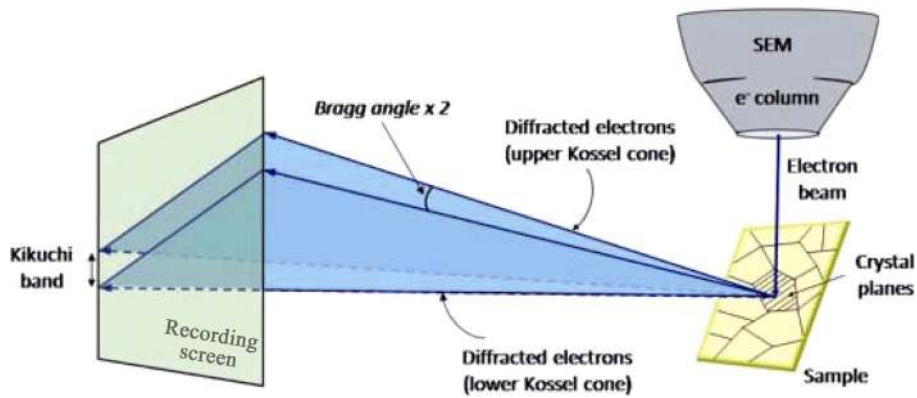


Fig. 5 Schematic of Kikuchi patterns obtained by EBSD. Reproduced with permission from Ref. [76], © Elsevier B.V. 2015.

higher spatial resolution than XRD as the electron beam can be focused.

Ni *et al.* [79] prepared textured h-BN matrix ceramics by slip casting and SPS using irregularly shaped and platelike h-BN powders as raw materials, respectively. Figures 6(a) and 6(b) give h-BN (002) pole figures on the surfaces perpendicular to the casting direction of slip casting green bodies obtained by XRD. The pole figure of the green body prepared using plate-like powders possesses stronger intensity in the center than the other, indicating that plate-like powders are more favorable to texture formation than irregular shaped powders. Figures 6(c) and 6(d) show fracture morphologies

of the surfaces parallel to the casting direction of sintered specimens, where the specimen prepared using plate-like powders is composed of plate-like grains with strong orientation, while that prepared using irregularly shaped powders mainly contains irregular shaped grains without orientation.

The numbers in pole figures of Fig. 6 are the multiples of a random distribution (*MRD*) defined as the volume fraction of the grains with certain orientation in the textured specimen divided by that in the homogeneous specimen, which can reflect texture degree. *MRD* = 1 and > 1 represent random orientation and preferred orientation, respectively. Higher *MRD* values represent

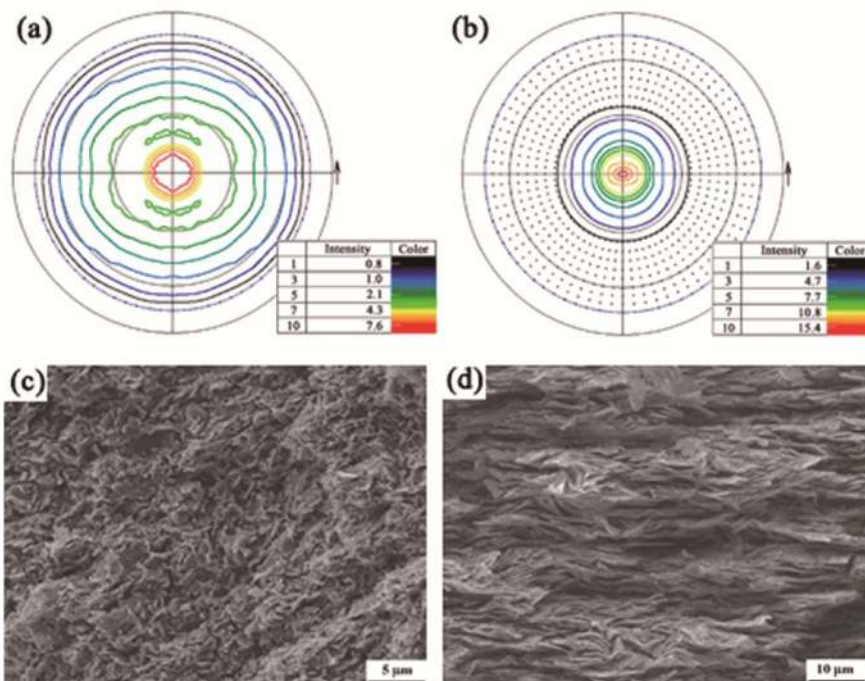


Fig. 6 h-BN (002) pole figures of surfaces perpendicular to the casting direction of green bodies and fracture morphologies of surfaces parallel to the casting direction of sintered specimens prepared using: (a, c) irregular shaped powders; (b, d) plate-like powders. Reproduced with permission from Ref. [79], © The American Ceramic Society 2011.

higher texture degree.

As the volume fraction of grains with a given orientation is proportional to the relative intensity of specific XRD peak, *MRD* values are usually obtained with XRD [79,80]. *MRD* values can be calculated by the March–Dollase function expressed as Eq. (2) [81–83]:

$$P_r(\eta) = (r^2 \cos^2 \eta + r^{-1} \sin^2 \eta)^{-3/2} \quad (2)$$

where r is the orientation parameter, which was defined as the ratio of the final specimen thickness after compaction to the initial specimen thickness in the original model. $r = 1$ for a homogeneous specimen and $r \rightarrow 0$ for a perfectly textured specimen. η is the tilt angle between the preferred orientation axis and the scattering vector. For the slip casting h-BN green bodies, the preferred orientation of the c -axis is parallel to the casting direction. If the green body possesses perfect texture microstructure, $r \rightarrow 0$, $P_r(\eta) \rightarrow \infty$ at $\eta = 0$ and $P_r(\eta) \rightarrow 0$ at $\eta = \pi/2$. So *MRD* values are the highest at the pole figure center in Fig. 6.

Figure 7 shows the Euler maps and inverse pole figure of textured α -Al₂O₃ ceramics prepared by magnetic alignment and pressureless sintering [84]. The orientation of each grain in Euler maps is represented by its color. The corresponding orientations of different colors are shown in Fig. 7(c). The inverse pole figure shows the orientation information on the cross-section perpendicular to the magnetic field direction. Combining Figs. 7(a), 7(b), and 7(c). (0 $\bar{1}$ 10), (0 $\bar{2}$ 10), and (0 $\bar{1}$ 00) crystal planes are mainly distributed on the surface parallel to the magnetic field direction, while (0001) plane is mainly distributed on the cross-section perpendicular to the magnetic field direction. The intensity around (0001) plane is the highest in Fig. 7(d), also indicating that (0001) plane is oriented perpendicular to the magnetic field direction, i.e., the c -axis is oriented parallel to the magnetic field direction.

2.2.2 Orientation factors calculated from XRD patterns

The formation of crystallographic texture in materials will correspond to the changing of relative intensities of XRD peaks, which are different from the standard data of the JCPDS card obtained from the powders of the specific material. Now several methods have been developed to calculate the orientation factors to quantitatively evaluate the texture degree, but we should determine the most suitable method according to the specific material.

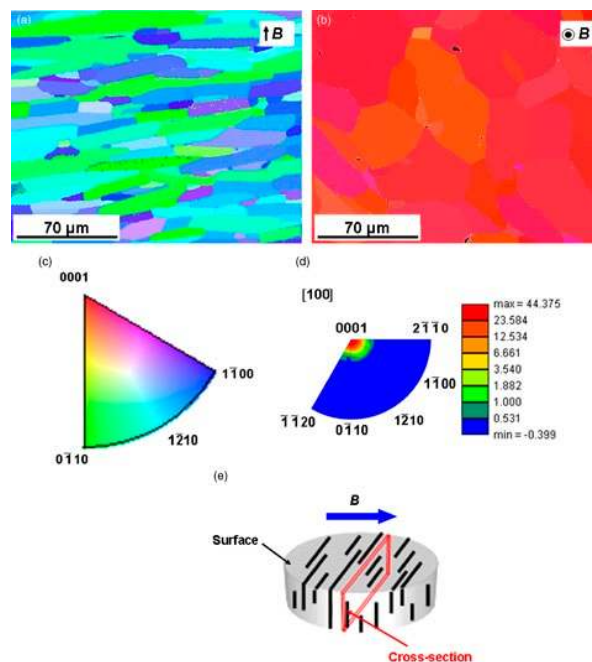


Fig. 7 Euler maps and inverse pole figure of textured α -Al₂O₃ ceramics prepared by magnetic alignment and pressureless sintering: (a, b) Euler maps of the surface and the cross-section of textured α -Al₂O₃ ceramics; (c) color map indicating the orientation in (a) and (b); (d) inverse pole figure for (b); (e) schematic diagram of the surface and the cross-section of textured α -Al₂O₃ ceramics. Reproduced with permission from Ref. [84], © The American Ceramic Society 2010.

(1) Lotgering factor f

The most widely used factor quantitatively characterizing texture degree is Lotgering factor f , which can be calculated according to Eq. (3) [40]:

$$f = \frac{P - P_0}{1 - P_0} \quad (3)$$

where P is the ratio of the diffraction peak intensity of the preferred orientation to all the diffraction peaks on the surface of textured sample, and P_0 represents that of the standard data. Obviously, $P \leq 1$. For the sample totally composed of randomly oriented grains, $P = P_0$ and $f = 0$. For one sample completely textured, that is, composed of grains with perfectly preferred orientation, $P = 1$, and $f = 1$. Lotgering factor reflects the fraction of the area with a specific preferred orientation. As for materials belonging to the hexagonal crystal lattice such as α -Al₂O₃, Si₃N₄, h-BN, and MAX phases, (00 l) peaks are the main diffraction peaks on the textured sample surface perpendicular to the preferred orientation of the c -axis, so $P = \sum I_{00l} / \sum I_{hkl}$, and the Lotgering factor is f_{00l} . If one sample belonging to

the hexagonal crystal lattice is totally textured, f_{00l} on that surface is 1. If the sample is totally composed of randomly oriented grains, f_{00l} on all surfaces of the sample is 0 [40]. Besides (00 l) planes, there are also some other planes can be used to calculate the Lotgering factor. For example, Zhang *et al.* [84] prepared textured α -Al₂O₃ by slip casting in a strong magnetic field parallel to the horizon, and the c -axis of α -Al₂O₃ was preferentially oriented parallel to the magnetic field. On the top surface of the sample, not (00 l) but (110) and (300) were the strong diffraction peaks, so $P = (I_{110} + I_{300}) / \sum I_{hkl}$.

(2) Index of orientation preference

Index of orientation preference (IOP) is another indicator that is usually used to characterize the texture degree of HPed h-BN ceramics. IOP values are calculated by Eq. (4) [1]:

$$IOP = \begin{cases} \frac{(I_{100} / I_{002})_{\text{perp}}}{(I'_{100} / I'_{002})_{\text{par}}}, (I_{100} / I_{002})_{\text{perp}} > (I'_{100} / I'_{002})_{\text{par}} \\ \frac{(I'_{100} / I'_{002})_{\text{par}}}{(I_{100} / I_{002})_{\text{perp}}}, (I_{100} / I_{002})_{\text{perp}} < (I'_{100} / I'_{002})_{\text{par}} \end{cases} \quad (4)$$

where I and I' are the corresponding diffraction intensities on the sample surface perpendicular and parallel to the external pressure, respectively. If the sample is composed of randomly oriented grains, $IOP = \pm 1$. If the c -axis of the sample is preferentially oriented parallel to the pressure direction, $IOP < -1$. If the c -axis of the sample is preferentially oriented perpendicular to the pressure, $IOP > 1$. Obviously, the larger absolute value of IOP indicates the more significant preferred orientation.

(3) Orientation factor P

Orientation factor P has been developed to characterize texture degree of α -Al₂O₃. On the textured α -Al₂O₃ sample surface perpendicular to the c -axis orientation, the texture degree can be calculated by Eq. (5) [69]:

$$P = I_{006} / (I_{006} + I_{110}) \quad (5)$$

For the standard α -Al₂O₃ powders, P is about 0.02 [85]. P can reach 1 for the perfectly textured α -Al₂O₃. The more closer P to 1 indicates the more significant texture of the sample.

3 Preparation of textured ceramics

In this part, three texturing techniques (hot working,

magnetic alignment, and TGG) commonly used for ceramic materials are reviewed. Their key technological parameters (the pressure for hot working, the magnetic intensity for magnetic alignment, the amount and morphology of template grains for TGG, etc.) influencing texture formation and properties of ceramics are analyzed.

Although there are some other methods, such as slip casting [79,86,87], extrusion [88–92], tape casting [93–98], can also be used to form texture microstructures in ceramics, they are usually used as aids to other techniques and not aimed at texture formation. For example, slip casting is usually conducted in magnetic fields to orientate ceramic grains with magnetic anisotropy [81,99–110], extrusion, and tape casting are usually used to align template grains for TGG [111–114]. So they are not discussed solely here.

3.1 Texturing techniques

3.1.1 Hot working

Hot working is to apply uniaxial pressure to align grains and form texture microstructures during high temperature sintering or heat treatment. As for metals, the external uniaxial pressure on the specimen can lead to grain boundary slip and dislocation movement inside grains [115,116]. As for ceramics, the uniaxial pressure can result in grain rotation and preferred orientation growth, forming texture microstructures [38,117]. External pressure can also promote densification of ceramic materials and reduce sintering temperature to a certain extent [118–121].

For ceramic materials belonging to the hexagonal crystal lattice, grains usually present plate-like or rod-like morphologies. It is easy for them to form texture by grain rotation during hot working. α -Al₂O₃, hexagonal boron nitride (h-BN), and MAX phases tend to grow along the a,b -axis, forming plate-like grains rotated with the c -axis parallel to the pressure direction (the c -axis oriented texture) [1,122,123]. Si₃N₄ and SiAlON incline to grow along the c -axis, forming rod-like grains rotated with the c -axis perpendicular to the pressure direction (the a,b -axis oriented texture), that is, rod-like grains tend to be arbitrarily oriented in the plane perpendicular to the pressure direction [6,124]. Besides these structural ceramics belonging to the hexagonal crystal lattice, hot working can also be used to prepare some textured piezoelectric ceramics with bismuth layered structure [125,126].

Hot working can be divided into hot-pressing (HP), hot-forging (HF), and sinter-forging (SF). HP is to sinter ceramics under uniaxial pressure. Spark plasma sintering (SPS), also called pulsed electric current sintering (PECS), also belongs to HP, and there is not only uniaxial pressure but also electric current applied to the specimen during SPS [40,126]. Figure 8 shows preparation and fracture morphologies of textured h-BN matrix ceramics by HP [127]. Plate-like h-BN grains can be rotated with the *c*-axis parallel to the pressure direction. Figure 8(a) shows the schematic diagram of the preparation process, where two plate-like specimens with their normals parallel and perpendicular to the HP direction and the crystal structure of h-BN are also shown. Figures 8(b) and 8(c) display fracture morphologies perpendicular and parallel to the HP direction of textured h-BN matrix ceramics, respectively, where plate-like h-BN grains are oriented perpendicular to the pressure direction. HF is a superplastic deformation technique to forge the sintered specimen by applying uniaxial pressure at high temperature. Figure 9 shows the preparation and fracture morphologies of textured Si₃N₄ matrix ceramics by hot working [23,128]. Figures 9(a) and 9(b) show two superplastic deformation techniques under simple compression (SC) and plane-strain compression (PSC). SC can generate

the *a,b*-axis oriented texture, which is the most common texture of Si₃N₄ ceramics containing rod-like grains. Xie *et al.* [129] prepared Si₃N₄-Si₂N₂O ceramics possessing the *c*-axis oriented texture with rod-like grains oriented in one direction by forging the specimen in a graphite channel die, that is, superplastic deformation under PSC. SF combines sintering and HF into one step [23,124,127,130]. Figure 9(c) shows the photographs of the Si₃N₄ green body after cold isostatic pressing (CIP) and the as-sintered Si₃N₄ specimen prepared by CIP and SF. Figures 9(d) and 9(e) show fracture morphologies of Si₃N₄ specimens prepared by HP and SF at 1900 °C for 3 h under 30 MPa, respectively. The SFed specimen possesses more significant texture microstructure than the HPed specimen, indicating SF is more favorable to texture formation than HP.

3.1.2 Magnetic alignment

Some materials possess magnetic anisotropy, i.e., the magnetic susceptibilities vary along different crystallographic directions, so they can be highly oriented by slip casting or gelcasting in magnetic fields [2,131]. For materials belonging to the hexagonal crystal lattice, such as α -Al₂O₃, Si₃N₄, and MAX phases, the anisotropy of magnetic susceptibilities $\Delta\chi$ can be expressed as Eq. (6) [132,133]:

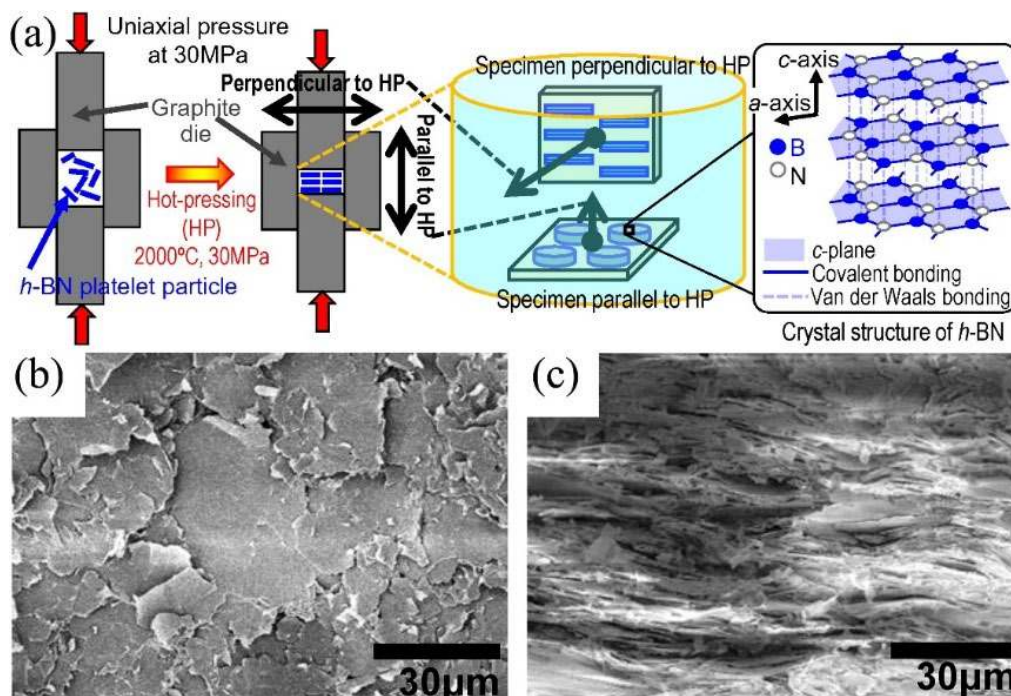


Fig. 8 Preparation and fracture morphologies of textured h-BN matrix ceramics prepared by HP: (a) schematic diagram of the preparation process; (b, c) fracture morphologies perpendicular and parallel to HP direction of textured h-BN ceramics sintered with 15 vol% Yb₂O₃-MgO additive, respectively. Reproduced with permission from Ref. [127], © Acta Materialia Inc. 2016.

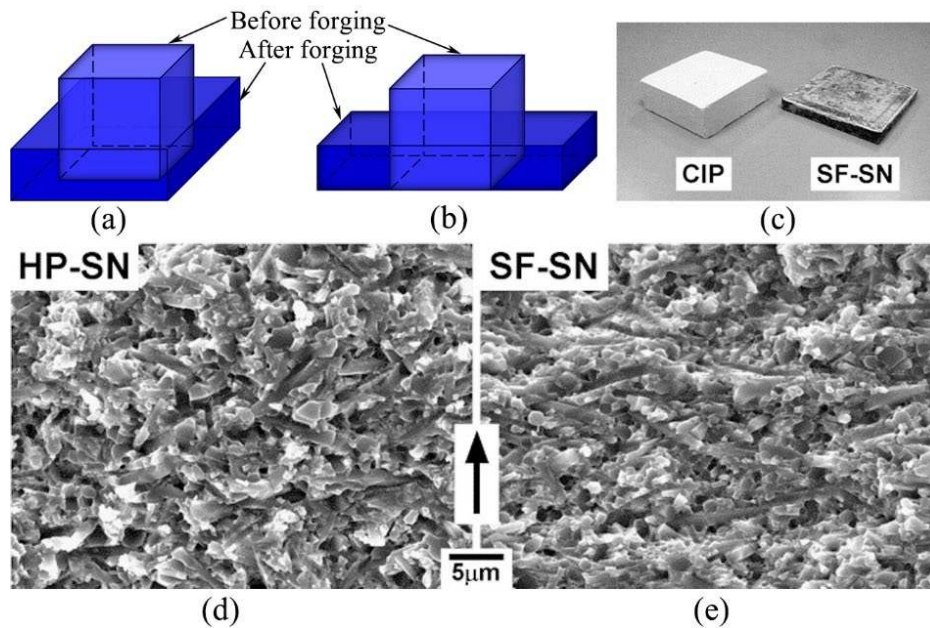


Fig. 9 Preparation and fracture morphologies of textured Si₃N₄ matrix ceramics prepared by hot working: (a, b) schematic diagram of superplastic deformation under SC and PSC, respectively; (c) CIPed Si₃N₄ green body (left) and SFed Si₃N₄ specimen (right); (d, e) fracture morphologies of Si₃N₄ specimens prepared by HP and SF, respectively. The arrow indicates crack propagation directions. Uniaxial pressure was applied vertically. Reproduced with permission from Ref. [128], © Elsevier Science Ltd. 2002.

$$\Delta\chi = \chi_c - \chi_{a,b} \tag{6}$$

where χ_c and $\chi_{a,b}$ are the magnetic susceptibilities along the c -axis and the a,b -axis, respectively.

When the material suspension is placed in a magnetic field, material particles tend to be rotated with the crystallographic axis possessing higher χ parallel to the magnetic field. The driving energy ΔE of rotation is given by Eq. (7) [132,133]:

$$\Delta E = \Delta\chi VB^2 / (2\mu_0) \tag{7}$$

where V is the volume of the material particle, B is the magnetic flux density, and μ_0 is the permeability in vacuum. The particle's thermal motion energy U can be described as Eq. (8) [134]:

$$U = 3nN_0k_B T \tag{8}$$

where n is the mole number of the material particle, N_0 is Avogadro constant, k_B is Boltzmann constant, and T is the temperature. When ΔE is higher than U , the particle can be rotated by the magnetic field. Figure 10 shows the schematic diagram of the effect of a magnetic field on the alignment of rod-like particles belonging to the hexagonal crystal lattice [2]. If the c -axis possesses higher χ , rod-like particles will be aligned parallel to the magnetic field. If the c -axis possessing lower χ , rod-like particles will be aligned

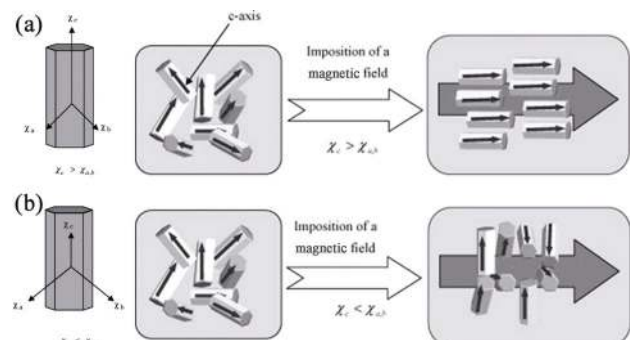


Fig. 10 Schematic diagram of the effect of a magnetic field on the alignment of rod-like particles belonging to the hexagonal crystal lattice: (a) $\chi_c > \chi_{a,b}$; (b) $\chi_c < \chi_{a,b}$. Reproduced with permission from Ref. [2], © Elsevier B.V. 2006.

perpendicular to the magnetic field. Obviously, the former case is more favorable to texture formation.

3.1.3 Templated grain growth

TGG is an effective technique to prepare textured ceramics by adding some template grains into matrix grains to promote preferred orientation growth. Grains with anisometric shape, such as plate-like and rod-like grains, are called template grains (or seeds). Grains with equiaxed shape are called matrix grains [32].

Firstly, template grains are dispersed into matrix grains. Then the green body is formed by tape casting or extrusion, where template grains are oriented and homogeneously distributed in matrix grains [23]. Figure 11 shows the schematic diagrams of preparing Si_3N_4 green body containing oriented $\beta\text{-Si}_3\text{N}_4$ seeds by extrusion and tape casting [23]. During subsequent liquid phase sintering (usually pressureless), materials of matrix grains are transported to template grains, leading to the preferred orientation growth of template grains, and finally textured ceramics can be obtained [3,32,135].

TGG has been widely used in the fabrication of textured piezoelectric ceramics with perovskite structure [136–140], because this kind of materials possesses very low crystallographic anisotropy (BaTiO_3 of the tetragonal crystal lattice has a small $c:a$ lattice constant ratio of about 1.007) and are very hard to be textured by hot working or magnetic alignment [141]. Based on TGG, reactive-templated grain growth (RTGG) was developed for these piezoelectric ceramics with high crystallographic symmetry. The difference between TGG and RTGG is that the target compound's precursors possessing anisometric shape are used and chemical reactions happen during RTGG [32,142–145]. Besides piezoelectric ceramics, TGG can also be applied to the texture formation of some structural ceramics, such as

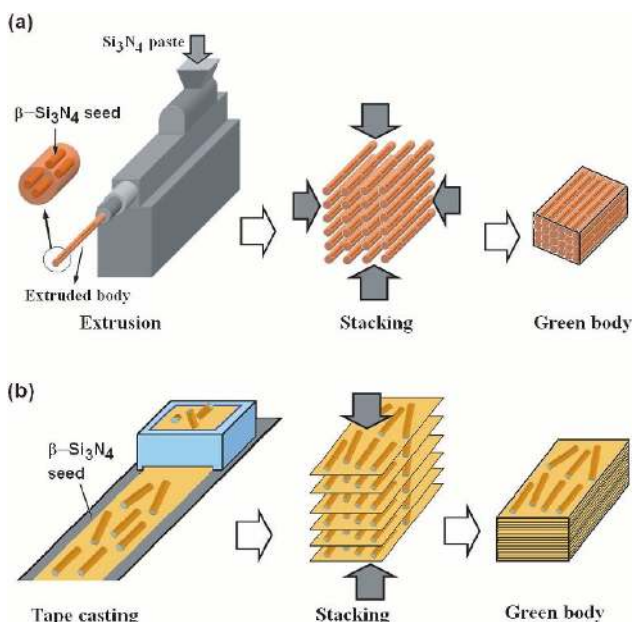


Fig. 11 Schematic diagrams of preparing Si_3N_4 green bodies containing oriented $\beta\text{-Si}_3\text{N}_4$ seeds by (a) extrusion and (b) tape casting. Reproduced with permission from Ref. [23], © National Institute for Materials Science 2008.

$\alpha\text{-Al}_2\text{O}_3$ and Si_3N_4 [3,146]. But it has seldom been used to prepare textured h-BN ceramics which are hard to be sintered without external pressure [147].

Although TGG is a very effective technique to prepare textured ceramics, densification is suppressed compared with using only equiaxed grains [3]. The hindrance of densification can be more serious for piezoelectric ceramics as the structure and chemical composition of template grains are usually different from that of matrix grains [148]. The above problems should be given prior consideration.

3.1.4 Other texturing techniques

In addition to these usual techniques discussed in Sections 2.1.1–2.1.3, some other techniques have also been developed to form texture microstructures in ceramics, such as freeze-casting which is usually used to prepare nacre bioinspired ceramics, as shown in Fig. 12 [53]. The general procedure is as follows. The starting colloids containing inorganic grains are frozen unidirectionally under the controlled temperature gradient. These inorganic grains grow between concentrated colloids. Then the solvent crystals are removed by sublimation, forming a layered porous scaffold composed of inorganic grains, where these pores are the replica of solvent crystals. The architecture of the scaffold can be adjusted by controlling the freezing kinetics of the solvent. Dense textured ceramics can be prepared by subsequent pressing and sintering. When the solvent is water, this method is also called ice-templating [53, 54,149,150].

Laser rapid solidification has been developed to form texture microstructures on material surfaces or prepare textured specimens of a few grams within several minutes [151]. Harimkar and Dahotre [152] prepared surface modified $\alpha\text{-Al}_2\text{O}_3$ ceramics by laser rapid solidification with the laser fluence ranging from 458 to 726 J/cm^2 . Faceted grains with $\{110\}$ and $\{211\}$ planes as surfaces are formed at the laser fluence of 573 J/cm^2 . Besides, the grain size of the modified surface increases as the laser fluence increases. Zhang *et al.* [151,153] conducted such research on texture formation of some ferroelectric ceramics including $\text{SrBi}_2\text{Nb}_2\text{O}_9$ and BaTi_2O_5 . Both of them tend to grow preferentially along $\langle 010 \rangle$ directions as their $\{010\}$ planes have the lowest atom density, forming the b -axis oriented ceramics composed of plate-like grains parallel to the laser incident direction.

Some additives are also favorable to texture formation

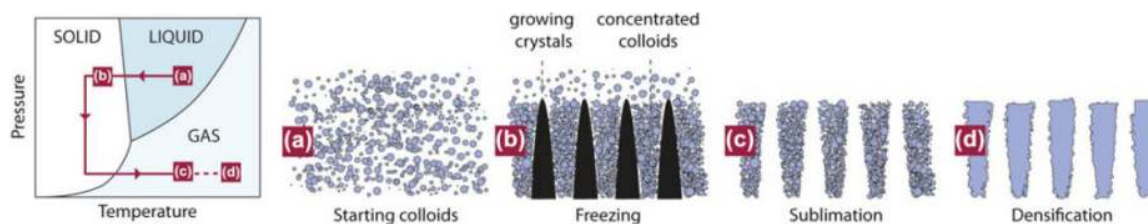


Fig. 12 Schematic diagram of freeze-casting. Reproduced with permission from Ref. [53], © Materials Research Society 2013.

of ceramics. Rutkowski *et al.* [154] prepared Si_3N_4 -graphene composites by HP. As the graphene addition increases from 0.5 to 10 wt%, the velocity anisotropy of longitudinal ultrasonic wave of the composites increases from 3% to 30%. Hubáček *et al.* [155] prepared textured BN-Cu composites by HP. The ultradispersed Cu in BN grains promotes the formation of donor-acceptor complexes between Cu electrons and π electrons of BN, activating lateral facets of BN and making them exposed to the uniaxial pressure, forming texture microstructure where the c -axis is oriented perpendicular to the pressure direction. But this effect is only remarkable when the Cu content is 1.5%–2.0%, where the index of orientation preference (IOP) reaches above 50.

3.2 Factors influencing texture formation

3.2.1 Raw materials

(1) Grain size of raw powders

Compared with coarse powders, fine powders are

more easily to be rotated under uniaxial pressure or magnetic fields. Zhang *et al.* [84] prepared textured $\alpha\text{-Al}_2\text{O}_3$ ceramics by slip casting in a horizontal magnetic field of 9.4 T using spherical raw powders with different sizes. The surface morphologies of these textured $\alpha\text{-Al}_2\text{O}_3$ specimens are shown in Fig. 13. The specimen prepared using the finest powders possesses the highest Lotgering factor, indicating it has the highest texture degree. That prepared by the coarsest powders has the lowest texture degree. Although that prepared using mixture of coarse and fine powders possesses grains with the highest aspect ratio, its Lotgering factor is not as high as that prepared by fine powders.

(2) Morphology of raw powders

Compared with equiaxed powders, anisometric powders are more easily to be oriented under uniaxial pressure. The SPSed Cr_2AlC specimen prepared by Duan *et al.* [123] using coarse equiaxed raw powders is composed of unoriented grains, while that prepared using as-milled fine plate-like powders is composed of oriented grains

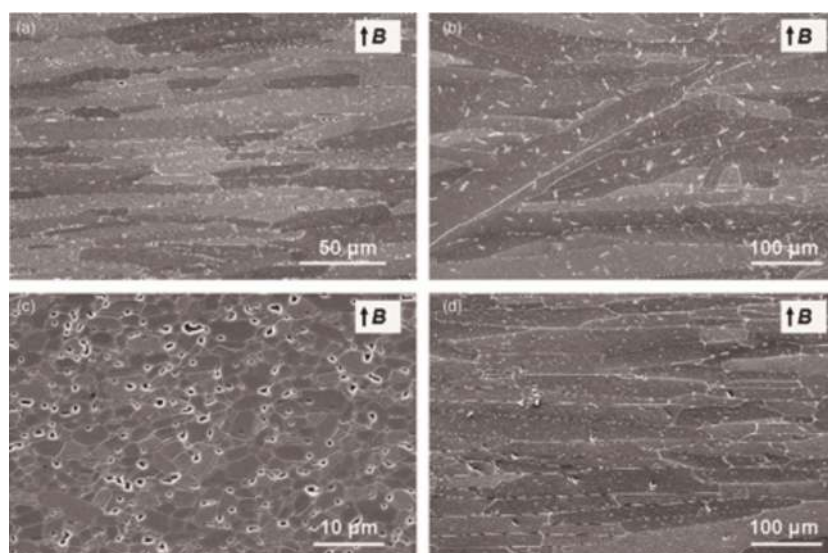


Fig. 13 Surface morphologies of textured $\alpha\text{-Al}_2\text{O}_3$ specimens prepared using raw powders with different grain sizes (μm): (a) 0.37; (b) 0.77; (c) 1.89; (d) 0.37 and 1.89 (7:3). The grain aspect ratios of each specimen are 4.52, 4.30, 1.86, and 5.45, respectively. The Lotgering factors $f_{110+300}$ of each specimen are 0.80, 0.18, 0.16, and 0.34, respectively. Reproduced with permission from Ref. [84], © The American Ceramic Society 2010.

with the c -axis parallel to the pressure direction, as shown in Fig. 14(a). Figures 14(b) and 14(c) show the Euler maps (orientation maps) of untextured and textured Cr_2AlC specimens obtained with EBSD using SEM, respectively. The grain color of the Euler map of the specimen sintered using fine plate-like powders is mostly green, and the grain size is very fine, indicating this specimen is composed of fine grains with strong orientation. While the specimen sintered using coarse equiaxed powders is composed of unoriented coarse grains. Figures 14(d) and 14(e) show the (0001) pole figure and inverse pole figure of the textured Cr_2AlC

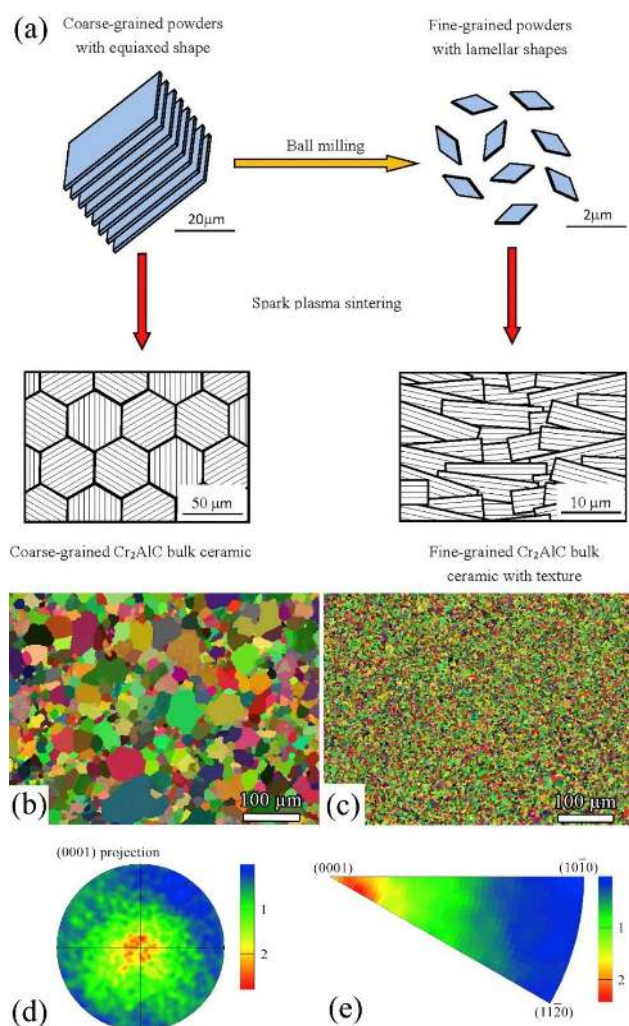


Fig. 14 Preparation and characterization of coarse-grained Cr_2AlC ceramic without texture and fine-grained Cr_2AlC ceramic with texture: (a) schematic diagrams of preparation; (b, c) Euler maps of untextured and textured specimens, respectively; (d, e) (0001) pole figure and inverse pole figure of textured Cr_2AlC specimen surface perpendicular to the sintering pressure, respectively. Reproduced with permission from Ref. [123], © Elsevier Ltd. 2014.

specimen surface perpendicular to the pressure direction obtained with EBSD using SEM. The intensity in the pole figure center is the highest, and the intensity around (0001) in the inverse pole figure is also the highest, indicating that Cr_2AlC grains are oriented with the c -axis parallel to the pressure direction.

(3) Morphology, size, and amount of template grains

As for TGG, the most important factor influencing texture formation is the characteristics of template grains. Takatori *et al.* [3] studied the effect of seeds on the texture formation of $\alpha\text{-Al}_2\text{O}_3$ ceramics prepared by tape casting and sintering. The results are shown in Table 1, where S, M, and L represent plate-like seeds of small, middle, and large size, respectively. Their average diameters are 0.51, 2.11, and 4.62 μm , and aspect ratios are 10, 25, and 25, respectively. The numbers following S, M, and L are the mass fraction of seeds. Euler maps of these specimens obtained by EBSD are shown in Fig. 15, where Tref indicates the specimen sintered with only equiaxed powders. Red grains in Euler maps indicate that the c -axis is oriented within 20° from the vertical direction. Table 1 reflects that finer powders are better to densification as they possess higher specific surface area favorable to sinterability. When the mass fraction of seeds is fixed at 5%, the M5 specimen possesses the highest Lotgering factor, which is consistent with Euler maps in Fig. 15, where the Euler map of the M5 specimen possesses more red grains than that of S5 and L5 specimens. M seeds have higher aspect ratio than S seeds and finer size than L seeds. Seeds with higher aspect ratio are more favorable to texture formation. Besides, when the mass fraction is fixed, M seeds are more favorable to texture formation than L seeds as there are more M seeds in the matrix grains of unit volume than L seeds, resulting in more effective preferred orientation growth. The M30 specimen possesses the highest Lotgering factor and the best c -axis oriented texture among M5, M30, and M60. Within a certain range, the more seeds added

Table 1 Effect of morphology, size, and amount of template grains on the texture degree of $\alpha\text{-Al}_2\text{O}_3$ ceramics [3]

Seed	Density (g/cm^3)	Lotgering factor $f_{006+1010}$
S5	3.95	0.22
M5	3.89	0.42
M30	3.89	0.69
M60	3.89	0.18
L5	3.87	0.22

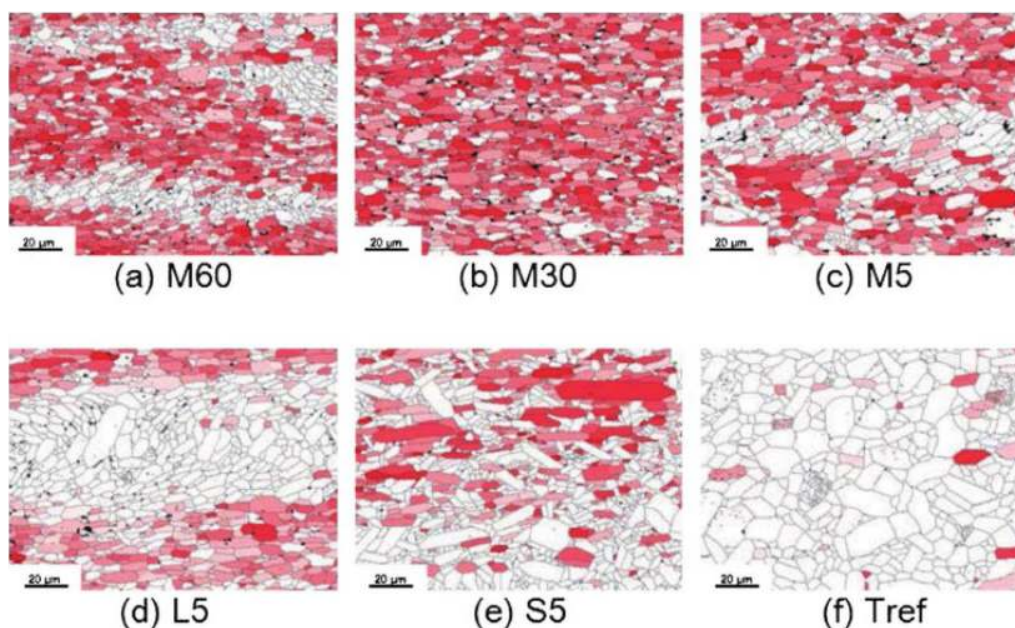


Fig. 15 Euler maps of α - Al_2O_3 specimens prepared using different seeds of different amount (the vertical direction in the maps corresponds to the stacking direction of plate-like seeds). Reproduced with permission from Ref. [3], © The Ceramic Society of Japan 2016.

into the matrix, the more significant texture can be formed. But when the amount surpasses a certain value, it will be unfavorable to texture formation. Because too many seeds mean the reduction of matrix grains that can be consumed to form anisometric grains and the decrease of distance between seeds, which tend to result in the formation of fine grains with low aspect ratio [22].

Template grains are usually aligned by the shearing force during tape casting or extrusion. So the alignment process is also important to texture formation. Taking tape casting as an example, besides higher solid content, higher carrier speed can also cause higher shear force. But if the speed is too high, template grains won't have enough time to be aligned [156]. Besides, the torque aligning template grains can be increased dramatically by dividing the flow into more flows during tape casting [157].

(4) Liquid phase sintering additives

Liquid phase sintering is a common method for preparation of ceramic materials. Liquid phase can provide a fast material diffusion path, so liquid phase sintering is more favorable to densification than solid state sintering [158–161]. Liquid phase can also provide an environment for grain rotation under uniaxial pressure, which is favorable to texture formation [129].

Seabaugh *et al.* [162] studied the amount of CaO– SiO_2 liquid phase sintering additive on the texture

formation of α - Al_2O_3 matrix ceramics. Morphologies of these specimens are shown in Fig. 16. The specimen prepared using 5 wt% liquid phase sintering additive has the most significant texture microstructure, because more liquid phase provides a faster material diffusion path for preferred orientation growth [147,163]. But the anisotropic grain growth results in a rigid network, causing grain interlocking, which is unfavorable to densification, as shown in Fig. 16 [162,164].

The wettability of liquid phase sintering additives to the matrix grains is also important to texture formation. It was demonstrated by Suzuki *et al.* [165] that TiO_2 and MgO are favorable to the sinterability and texture formation of α - Al_2O_3 , while Y_2O_3 stabilized ZrO_2 is not. Kusunose and Sekino [127] proved that the composite additives of a rare-earth oxide and an alkaline-earth oxide exhibit better wettability to h-BN grains and are better to texture formation and directional thermal conduction performance of HPed h-BN matrix ceramics than the simple additive of an alkaline-earth oxide. So appropriate additives should be chosen for specific materials.

(5) Suspension viscosity

Magnetic alignment is usually combined with slip casting, gelcasting, or electrophoretic deposition, so the suspension viscosity can significantly affect grain rotation in the magnetic field. Low suspension viscosity benefits its flowability, thus has a positive

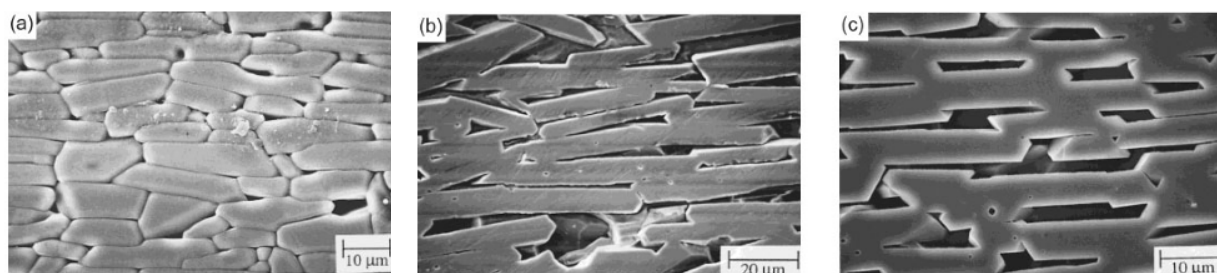


Fig. 16 Morphologies of liquid phase sintered α -Al₂O₃ specimens using 4 wt% oriented template grains and different amount of CaO–SiO₂ additive: (a) 0.5 wt%, (b) 1 wt%, (c) 5 wt%. Reproduced with permission from Ref. [162], © John Wiley and Sons 2005.

effect on the alignment of particles. Low viscosity is also good for densification [132]. Suzuki *et al.* [166] conducted such research on textured α -Al₂O₃ ceramics prepared by slip casting in a magnetic field of 10 T and sintering at 1600 °C for 2 h. The results are shown in Table 2. Moderate dispersant amount and low solid content result in low suspension viscosity and high texture degree.

However, gelcasting seems to be more effective for suspension with high solid content, which was proposed by Szudarska *et al.* [167]. After gelcasting in a magnetic field of 12 T and subsequent sintering at 1600 °C for 2 h, the textured α -Al₂O₃ specimen prepared from suspension with 45 vol% solid possesses higher texture degree than that prepared from suspension with 40 and 50 vol% solid, though its viscosity is in the medium of the three. Besides, high solid content is favorable to the quality of the green body. So the solid content should be decided by considering both the viscosity and the quality of the green body [168].

3.2.2 Preparation process factors

(1) Sintering temperature

High sintering temperature is generally beneficial to

Table 2 Effect of dispersant amount and solid content on the viscosity of suspensions and texture degree of sintered α -Al₂O₃ specimens [166]

Solid content (vol%)	Dispersant amount (wt%)	Viscosity (mPa·s, measured at a shear rate of 100 s ⁻¹)	Orientation factor <i>P</i>
30	0.44	60.07	0.04
	0.50	14.31	0.98
	0.58	10.38	0.99
	1.20	17.45	0.93
30	—	28.59	0.94
40	—	200.51	0.91
45	—	334.48	0.03

densification and texture formation of ceramics. Uchikoshi *et al.* [169] prepared textured α -Al₂O₃ ceramics by electrophoretic deposition in a magnetic field of 10 T and subsequent sintering. As the sintering temperature increases from 800 to 1600 °C, the orientation factor $I_{006}/(I_{006}+I_{110})$ increases from 0.08 to 0.96. Meanwhile, the relative density also increases from 59% to 97%.

Takatori *et al.* [3] studied the effect of sintering temperature on texture formation of α -Al₂O₃ ceramics prepared by TGG. Fracture morphologies of α -Al₂O₃ specimens sintered at different temperatures using 30 and 100 wt% seeds with average diameter of 2.11 μ m and aspect ratio of 25 (marked as M30 and M100) are shown in Fig. 17. When the sintering temperature is 1400, 1600, and 1700 °C, the Lotgering factors f_{006} of M30 are 0.36, 0.55, and 0.69, respectively, and that of M100 are 0.26, 0.41, and 0.15, respectively. For M30, the preferred orientation growth and densification are both promoted by high sintering temperature. For M100, too high sintering temperature may be not favorable to texture formation.

(2) Sintering pressure

Duan *et al.* [1] and Xue *et al.* [170] studied the effect of sintering pressure on the texture degree of h-BN matrix ceramics prepared by HP, as shown in Table 3. For plate-like h-BN powders, as the pressure increases, they are more easily to be rotated with the *c*-axis parallel to the pressure direction, causing higher texture degree ($IOP < 0$). But when h-BN raw powders possess irregular shape, the uniaxial pressure is not very high, and there is no liquid phase sintering additive used, the pressure can promote grain contact, causing the preferred orientation growth of plate-like grains along the pressure direction ($IOP > 0$).

The HPed h-BN (002) pole figures of textured h-BN–mullite composites prepared under different sintering pressure are shown in Figs. 18(a), 18(c), and 18(e). As

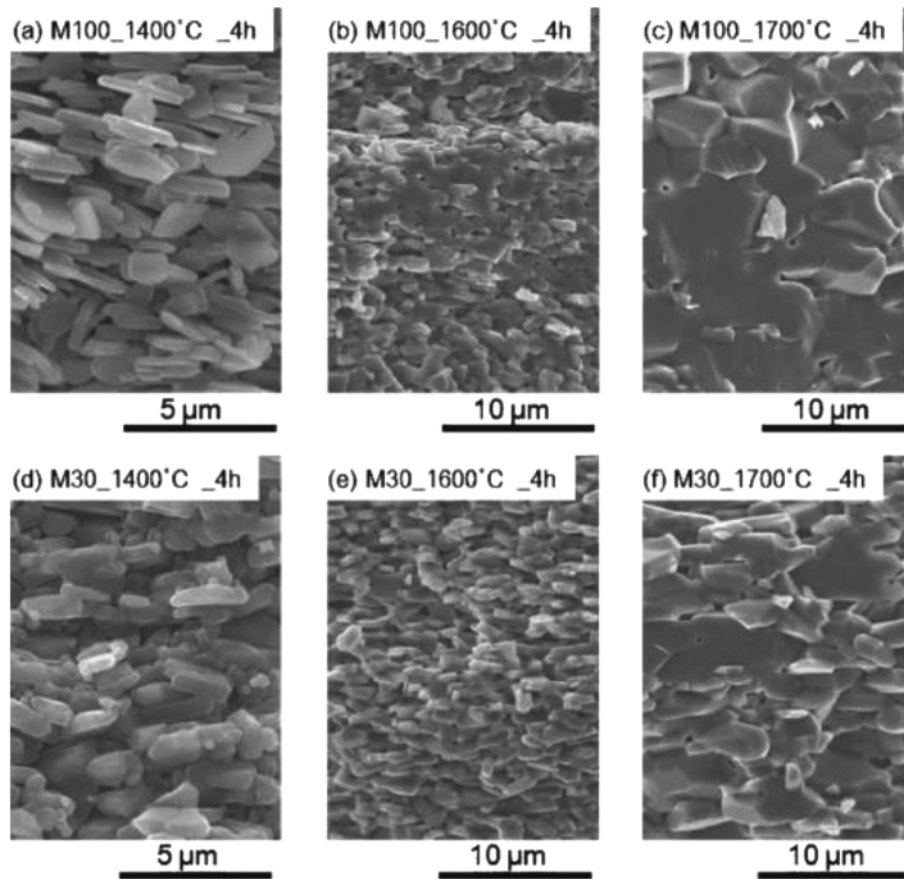


Fig. 17 Fracture morphologies of textured α -Al₂O₃ specimens with different amount of seeds sintered at different temperatures for 4 h in air. Reproduced with permission from Ref. [3], © The Ceramic Society of Japan 2016.

Table 3 Effect of sintering pressure on the texture degree of HPed h-BN matrix ceramics

Researchers	Raw materials	Sintering pressure	IOP value
Xue <i>et al.</i> [170]	Irregular shaped h-BN	15 MPa	11.5
		30 MPa	27.2
		45 MPa	20.6
		60 MPa	10.9
Duan <i>et al.</i> [1]	Plate-like h-BN + 20 wt% mullite	10 MPa	-178
		20 MPa	-350
		30 MPa	-2160

the sintering pressure increases, the intensity at the center of the pole figure increases, indicating that stronger texture has been formed by higher pressure. Figures 18(b), 18(d), and 18(f) show the relative volume fraction distribution of h-BN grains in textured composites along the (0, ϕ , 5) direction obtained from orientation distribution function (ODF), where ϕ represents the deviation angle of the *c*-axis from the ideal orientation (the pressure direction). As the sintering pressure increases, more h-BN grains are oriented with

smaller deviation angles, also demonstrating the positive effect of sintering pressure on texture formation.

(3) Magnetic intensity

According to Eq. (7), for these materials possessing anisotropic magnetic susceptibility, the stronger the applied magnetic field is, the higher energy materials possess to be rotated to the easy magnetization direction. Uchikoshi *et al.* [132] studied the effect of magnetic intensity on the texture degree of α -Al₂O₃ ceramics prepared by magnetic alignment and sintering. The texture degree increases with the increase of magnetic field intensity, as shown in Fig. 19.

Terada *et al.* [67] studied texture development of α -Al₂O₃ suspension in a changing magnetic field by *in situ* ND. The schematic diagram of the experimental apparatus is shown in Fig. 20(a). The magnetic field dependence of diffraction intensities of different crystal planes of α -Al₂O₃ is shown in Fig. 20(b). As the magnetic intensity increases, the diffraction intensity of (006) decreases, indicating that α -Al₂O₃ particles tend to be aligned with the *c*-axis parallel to the magnetic

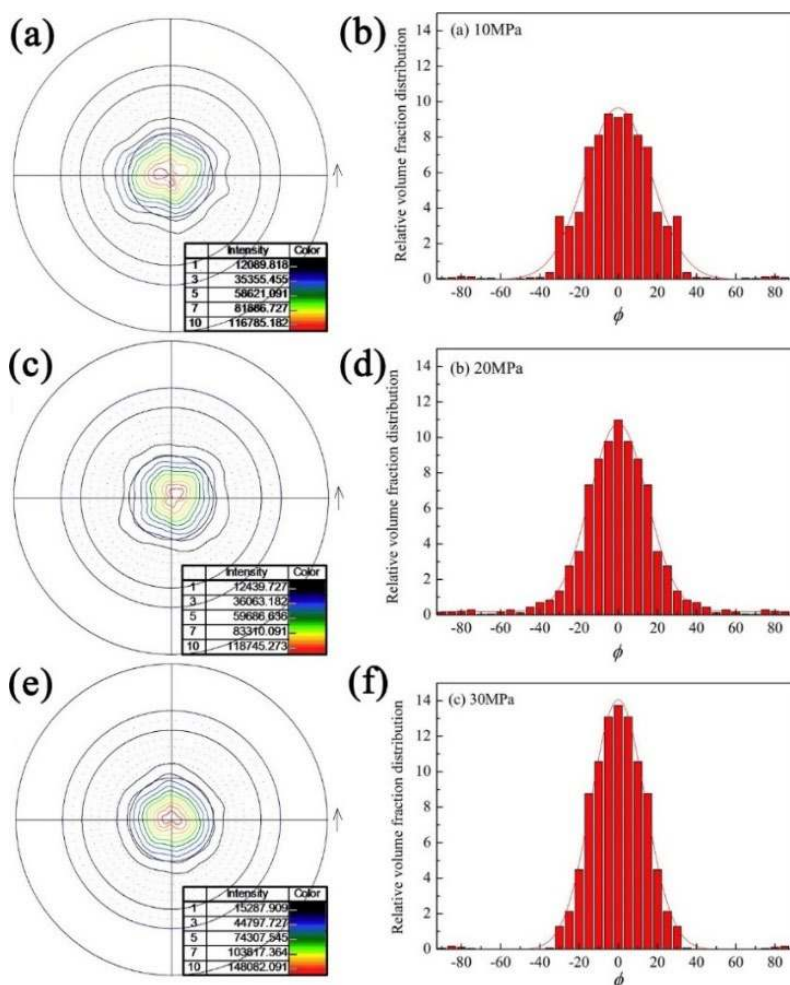


Fig. 18 (002) pole figures and relative volume fraction distribution of h-BN grains in textured h-BN matrix ceramics prepared under different sintering pressure: (a, b) 10 MPa; (c, d) 20 MPa; (e, f) 30 MPa. Reproduced with permission from Ref. [1], © Acta Materialia Inc. 2012.

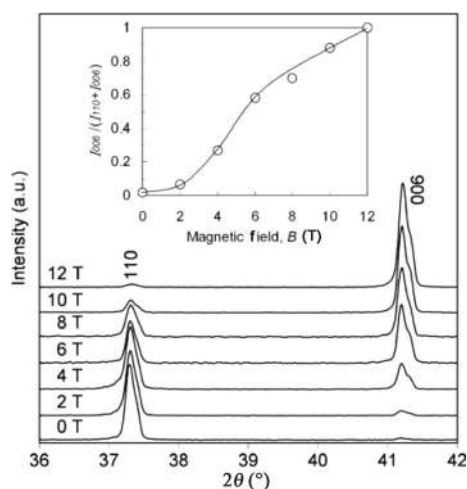


Fig. 19 Effect of magnetic intensity on (110) and (006) diffraction peak intensities and orientation factors of α -Al₂O₃ specimen surfaces perpendicular to the magnetic field. Reproduced with permission from Ref. [132], © Springer Science+Business Media, LLC 2006.

field. Then the magnetic intensity and sintering temperature dependence of the orientation parameter r of the sintered α -Al₂O₃ ceramics is also studied [64]. Figure 21(a) shows the measuring apparatus, where the double arrow indicates the magnetic field direction parallel to the scattering vector. The specimen holder was horizontally rotated, and the scattering angle, 2θ , was changed to measure diffraction intensities of different crystal planes. The measuring result is shown in Fig. 21(b). As the magnetic intensity and sintering temperature increase, the orientation parameter r of the sintered α -Al₂O₃ specimen is more close to 0, indicating the formation of stronger texture.

As for gelcasting in magnetic fields, the magnetic field exposure time and the idle time between the moment of adding the initiator into the suspension and the moment when viscosity increases rapidly due to the polymerisation can also influence texture formation. It

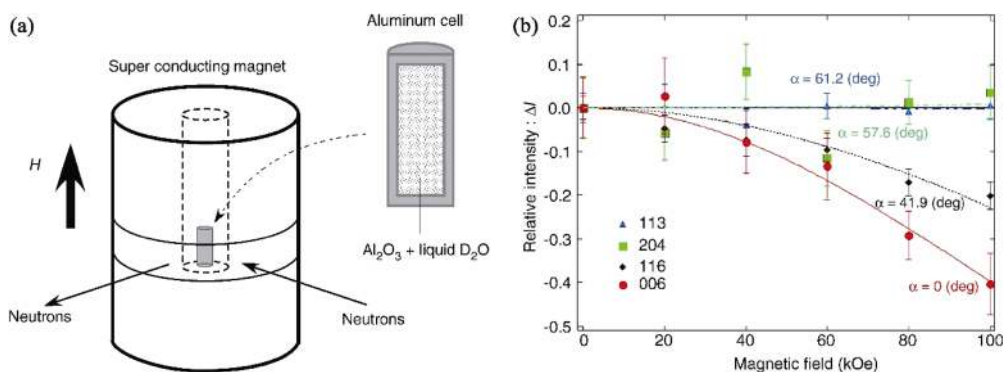


Fig. 20 Characterization of texture development of α -Al₂O₃ suspension in a changing magnetic field: (a) schematic diagram of the experimental apparatus; (b) magnetic field dependence of diffraction intensities of different crystal planes of α -Al₂O₃. Reproduced with permission from Ref. [67], © American Institute of Physics 2008.

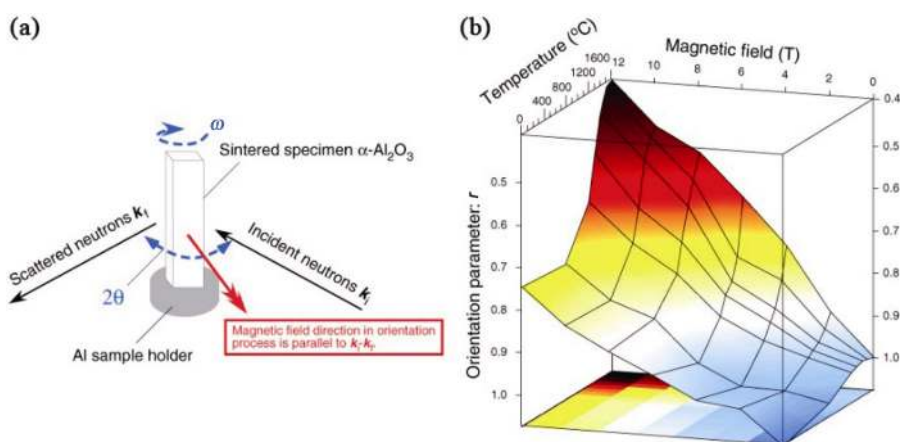


Fig. 21 Magnetic intensity and sintering temperature dependence of the orientation parameter r of the sintered α -Al₂O₃: (a) schematic diagram of the experimental apparatus; (b) measuring result. Reproduced with permission from Ref. [64], © IOP Publishing Ltd. 2009.

has been confirmed that short idle time and long magnetic field exposure time are favorable to the orientation of particles during gelcasting. The idle time can be controlled by adjusting the amount of catalyst and initiator [167,171].

3.3 Comparisons of texturing techniques

Each texturing technique has its own advantages and disadvantages. Hot working is usually used to prepare textured ceramics with anisometric grains, but it is not useful to prepare textured materials with equiaxed grains. Magnetic alignment can align all materials possessing crystal magnetic anisotropy, but many ceramic materials belong to weak magnetic substances. So strong magnetic fields up to 10 T are needed to texture them. TGG is low-cost and can form significant texture microstructures. But TGG green bodies usually need to go through pressureless sintering, so it is hard to densify some materials hard to be sintered such as

h-BN. The difference between template grains and matrix grains can also suppress densification. The characteristics of these techniques are listed in Table 4.

4 Typical textured structural ceramics and their anisotropy

Mechanical properties along specific directions of structural ceramics can be significantly improved by texturing so that they can be applied to more harsh service environments. In this part, several typical textured structural ceramics including α -Al₂O₃ and α -Al₂O₃ related nacre bioinspired ceramics, Si₃N₄ and SiAlON, h-BN, MB₂ matrix ultra-high temperature ceramics, MAX phases and some other ceramics and their anisotropic properties are discussed. α -Al₂O₃ is one of the most common structural ceramics and has been widely used [172–177]. Si₃N₄ and SiAlON are

Table 4 Comparisons of texturing techniques

Techniques	Advantages	Disadvantages	Materials requirements	Typical materials
Hot working	Accelerate mass transfer process and densification	High cost High energy consumption	Materials with anisometric crystal structure	α -Al ₂ O ₃ h-BN Si ₃ N ₄ MAX phases
Magnetic alignment	All grains can be aligned	Strong magnetic fields are needed	Materials possessing magnetic anisotropy	α -Al ₂ O ₃ Si ₃ N ₄ MAX phases
TGG	Simple process Low-cost	Densification is hard	Materials easy to be sintered	α -Al ₂ O ₃ Si ₃ N ₄ Piezoelectric ceramics

usually used to fabricate whisker-reinforced ceramics due to their excellent mechanical properties [178,179]. The lamellar structure of h-BN enables it to be used as directional thermal conductive materials [147,180–182]. MB₂ matrix ultra-high temperature ceramics have extreme high temperature that allows them to be used in extreme environments [183,184]. MAX phases have been widely studied recently due to their excellent properties combining that of metals and ceramics [180, 185–189].

4.1 α -Al₂O₃ and α -Al₂O₃ related nacre bioinspired ceramics

4.1.1 α -Al₂O₃

Alumina is present in nature with a variety of phases, in which α -Al₂O₃ also called corundum belongs to the hexagonal crystal lattice [190,191]. α -Al₂O₃ is thermodynamically stable and has many excellent properties such as high strength and hardness, low dielectric loss, good insulation performance, good resistance to oxidation at high temperature, outstanding biocompatibility, etc. It has been widely used in electrical, mechanical, biomedical, chemical, optical, and refractory fields [172–177].

α -Al₂O₃ possesses a slight crystal magnetic anisotropy,

$\Delta\chi = \chi_c - \chi_{a,b} = 7.1 \times 10^{-8} \text{ emu} \cdot \text{mol}^{-1}$, so the *c*-axis of α -Al₂O₃ can be aligned parallel to the magnetic field direction. But the $\Delta\chi$ is so low that strong magnetic fields are needed to form preferred grain orientation [64]. Figure 22 shows the polished surfaces of α -Al₂O₃ ceramics prepared with and without the magnetic field [69]. Plate-like α -Al₂O₃ grains are oriented with the *c*-axis parallel to the magnetic field. Due to the anisometric shape of α -Al₂O₃ grains, hot working and TGG can also be used to prepared textured α -Al₂O₃ ceramics [135,177].

During the preparation of textured α -Al₂O₃ ceramics by TGG, the increase of seed amount can result in the decrease of grain size in the sintered specimen, which leads to the increase of flexural strength [192]. Fine seeds are more favorable to flexural strength than coarse seeds due to the same reason [193]. It is worth noting that high seed amount is not always favorable to texture formation of α -Al₂O₃ ceramics, as discussed in Section 3.2.1.

Table 5 lists the mechanical and thermal properties of textured α -Al₂O₃ matrix ceramics prepared by different methods. Textured α -Al₂O₃ ceramics usually show better mechanical properties (flexural strength and fracture toughness) with the load parallel to the *c*-axis orientation than perpendicular to the *c*-axis

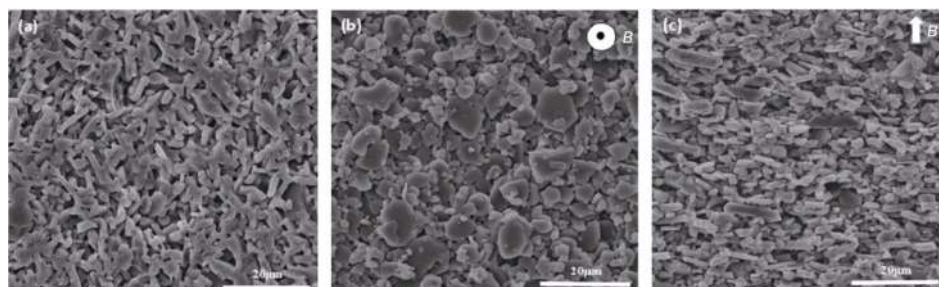


Fig. 22 Polished surfaces of untextured and textured α -Al₂O₃ specimens: (a) untextured specimen; (b, c) textured specimen surfaces perpendicular and parallel to the magnetic field direction, respectively. Reproduced with permission from Ref. [69], © WILEY - VCH Verlag GmbH & Co. KGaA, Weinheim 2015.

Table 5 Properties of textured Al₂O₃ matrix ceramics

Researchers	Raw materials	Preparation methods	Relative density (%)	Orientation factor	Flexural strength (MPa)	Fracture toughness (MPa·m ^{1/2})	Thermal conductivity (W/(m·K))
Yang <i>et al.</i> [69]	Irregular shaped α-Al ₂ O ₃	Pressureless sintering (1600 °C)	66.34	$P = 0.07$	55.41	—	—
		Magnetic alignment & pressureless sintering (1500 °C)	64.00	$P = 0.68$	95.66 (<i>// c-axis</i>) 80.21 (<i>⊥ c-axis</i>)	—	—
		Magnetic alignment & pressureless sintering (1550 °C)	64.11	$P = 0.68$	97.65 (<i>// c-axis</i>) 80.21 (<i>⊥ c-axis</i>)	—	—
		Magnetic alignment & pressureless sintering (1600 °C)	66.29	$P = 0.70$	105.63 (<i>// c-axis</i>) 85.96 (<i>⊥ c-axis</i>)	—	—
Honda <i>et al.</i> [40]	Plate-like α-Al ₂ O ₃	SPS (1300 °C)	67.56	$f_{001} = 0.31$	155.17 (<i>// c-axis</i>) 46.73 (<i>⊥ c-axis</i>)	—	3.4 (<i>// c-axis</i>) 10.4 (<i>⊥ c-axis</i>)
		SPS (1400 °C)	80.84	$f_{001} = 0.50$	403.65 (<i>// c-axis</i>) 217.84 (<i>⊥ c-axis</i>)	—	10.5 (<i>// c-axis</i>) 22.3 (<i>⊥ c-axis</i>)
		SPS (1500 °C)	90.47	$f_{001} = 0.51$	373.68 (<i>// c-axis</i>) 251.08 (<i>⊥ c-axis</i>)	—	11.0 (<i>// c-axis</i>) 22.6 (<i>⊥ c-axis</i>)
		SPS (1600 °C)	95.06	$f_{001} = 0.71$	416.91 (<i>// c-axis</i>) 253.89 (<i>⊥ c-axis</i>)	—	30.4 (<i>// c-axis</i>) 35.7 (<i>⊥ c-axis</i>)
		SPS (1650 °C)	97.17	$f_{001} = 0.85$	437.80 (<i>// c-axis</i>) 256.16 (<i>⊥ c-axis</i>)	—	31.6 (<i>// c-axis</i>) 35.3 (<i>⊥ c-axis</i>)
Nishimura <i>et al.</i> [122]	Platelike α-Al ₂ O ₃ + 10 wt% fine α-Al ₂ O ₃	SPS	95	$f_{001} = 0.34$	338.46 (<i>// c-axis</i>) 151.58 (<i>⊥ c-axis</i>)	4.2 (<i>// c-axis</i>) 1.8 (<i>⊥ c-axis</i>)	36 (<i>// c-axis</i>) 39 (<i>⊥ c-axis</i>)
Carisey <i>et al.</i> [192]	α-Al ₂ O ₃ + 0–10 wt% plate-like α-Al ₂ O ₃ seeds with size of 3–7 μm and 10–15 μm	TGG & pressureless sintering (0 wt% seeds)	—	—	383.62 (<i>// c-axis</i>) 371.16 (<i>⊥ c-axis</i>)	6.47 (<i>// c-axis</i>) 4.86 (<i>⊥ c-axis</i>)	—
		TGG & pressureless sintering (5 wt% 3–7 μm seeds)	—	—	471.52 (<i>// c-axis</i>) 402.96 (<i>⊥ c-axis</i>)	5.45 (<i>// c-axis</i>) 5.28 (<i>⊥ c-axis</i>)	—
		TGG & pressureless sintering (5 wt% 10–15 μm seeds)	—	—	432.90 (<i>// c-axis</i>) 357.26 (<i>⊥ c-axis</i>)	4.57 (<i>// c-axis</i>) 5.47 (<i>⊥ c-axis</i>)	—
		TGG & pressureless sintering (10 wt% 3–7 μm seeds)	—	—	465.87 (<i>// c-axis</i>) 444.60 (<i>⊥ c-axis</i>)	5.25 (<i>// c-axis</i>) 4.89 (<i>⊥ c-axis</i>)	—
		TGG & pressureless sintering (10 wt% 10–15 μm seeds)	—	—	436.67 (<i>// c-axis</i>) 386.97 (<i>⊥ c-axis</i>)	5.15 (<i>// c-axis</i>) 5.39 (<i>⊥ c-axis</i>)	—
Pavlacka and Messing [193]	α-Al ₂ O ₃ + 0.14 wt% (CaO + SiO ₂) + 1–15 vol% plate-like α-Al ₂ O ₃	TGG & pressureless sintering (1 vol% seeds)	98.20	$r = 0.13$	305 (<i>// c-axis</i>) 316 (<i>⊥ c-axis</i>)	3.05 (<i>// c-axis</i>) 3.09 (<i>⊥ c-axis</i>)	—
		TGG & pressureless sintering (5 vol% seeds)	97.38	$r = 0.14$	417 (<i>// c-axis</i>) 421 (<i>⊥ c-axis</i>)	3.03 (<i>// c-axis</i>) 3.93 (<i>⊥ c-axis</i>)	—
		TGG & pressureless sintering (10 vol% seeds)	94.49	$r = 0.16$	420 (<i>// c-axis</i>) 384 (<i>⊥ c-axis</i>)	3.11 (<i>// c-axis</i>) 3.90 (<i>⊥ c-axis</i>)	—
		TGG & pressureless sintering (15 vol% seeds)	93.11	$r = 0.17$	436 (<i>// c-axis</i>) 511 (<i>⊥ c-axis</i>)	2.55 (<i>// c-axis</i>) 4.58 (<i>⊥ c-axis</i>)	—

orientation. When the loading direction is parallel to the *c*-axis orientation, there are a large number of grains playing the role of load transfer, and the crack is transferred to the direction along the interfaces of these plate-like grains. So the crack needs to take longer paths to make fracture occur, and more energy needs to be consumed. When the external stress is perpendicular to the *c*-axis orientation, grains playing the role of load transfer are less, and the crack easily propagates along the interfaces of these plate-like grains, making the fracture occur easily than the former [40,194]. However, in the research of Pavlacka and Messing [193], the

fracture toughness parallel to the *c*-axis orientation is surprisingly lower than that perpendicular to the *c*-axis orientation. Because the crack goes through the thickness parallel to the *c*-axis orientation (the smaller dimension of textured grains), which makes them more difficult to bridge effectively. So which direction possesses higher toughness is determined by whether crack deflection or bridging is the dominant toughening mechanism.

The thermal conductivity of textured α-Al₂O₃ ceramics parallel to the *c*-axis orientation is lower than that perpendicular to the *c*-axis orientation [40,122]. There are more grain boundaries and defects along the *c*-axis

orientation, so phonons are scattered more seriously, resulting in the lower thermal conductivity. α -Al₂O₃ ceramics sintered at higher temperature have higher and more anisotropic flexural strength, because high sintering temperature is beneficial to densification and texture formation. But the anisotropy of thermal conductivities may not be promoted by high sintering temperature. Because the decrease of porosity combines grains more closely, thermal conductivities increase along both directions [40,69].

Besides α -Al₂O₃ bulk ceramics with the single texture microstructure, multilayered α -Al₂O₃ ceramics with alternate orientations can be prepared by electrophoretic deposition in a magnetic field and alternately changing the included angle Φ_{B-E} between the electric field and the magnetic field. Figure 23 shows the preparation and microstructures of multilayered α -Al₂O₃ ceramics by electrophoretic deposition in a magnetic field [195]. When the loading direction is perpendicular to the substrate, the crack propagates along grain boundaries, causing a complete intergranular fracture. When the loading direction is parallel to the substrate, the crack propagates through grains, causing an intragranular fracture. As a result, both the flexural strength and fracture toughness for the crack-propagation direction parallel to the substrate (415 MPa, 4.99 MPa·m^{1/2}) are higher than that perpendicular to the substrate (303 MPa, 3.65 MPa·m^{1/2}) [195,196]. The texture types of multilayered α -Al₂O₃ ceramics can be adjusted by controlling the electric field and the magnetic field. For example, the multilayered α -Al₂O₃ specimen with oriented and unoriented layers alternatively arranged

can be prepared by alternate electrophoretic deposition in and out of a magnetic field [197].

4.1.2 α -Al₂O₃ related nacre bioinspired ceramics

Mother-of-pearl (nacre) has excellent mechanical properties due to its specific brick-and-mortar structure. It is composed of 95 wt% plate-like aragonite whose chemical component is calcium carbonate and 5 wt% organic matrix whose components are proteins and polysaccharides. Polysaccharides are composed of cellulose and chitin [45,46,198]. Figure 24 shows the five scale levels of abalone nacre [46]. At level II, there are many nanoasperities on the surfaces of aragonite tiles. Although the ceramic content is very high, nacre possesses a fracture work 3000 times higher than aragonite. Its main toughening mechanisms are crack deflection at the interfaces between aragonite tiles and organic matrix, fiber pull-out of aragonite tiles, and crack bridging owing to organic fibers [199]. Besides, these organic layers can act as viscoelastic glue to prevent the deformation between aragonite platelets [57].

Much research has been carried out to prepare materials possessing the hierarchical structure similar to nacre by freeze-casting. Bouville *et al.* [55] prepared nacre-like α -Al₂O₃ ceramics by ice-templating and subsequent pressing and sintering. The α -Al₂O₃ specimen possesses a long-range ordered structure as shown in Fig. 25(a). It is composed of 98.5 vol% α -Al₂O₃, 1.3 vol% silica, and 0.2 vol% calcia. The dimension of its closely packed ceramic platelets is identical to that of nacre. There are many nanoasperities on the surfaces

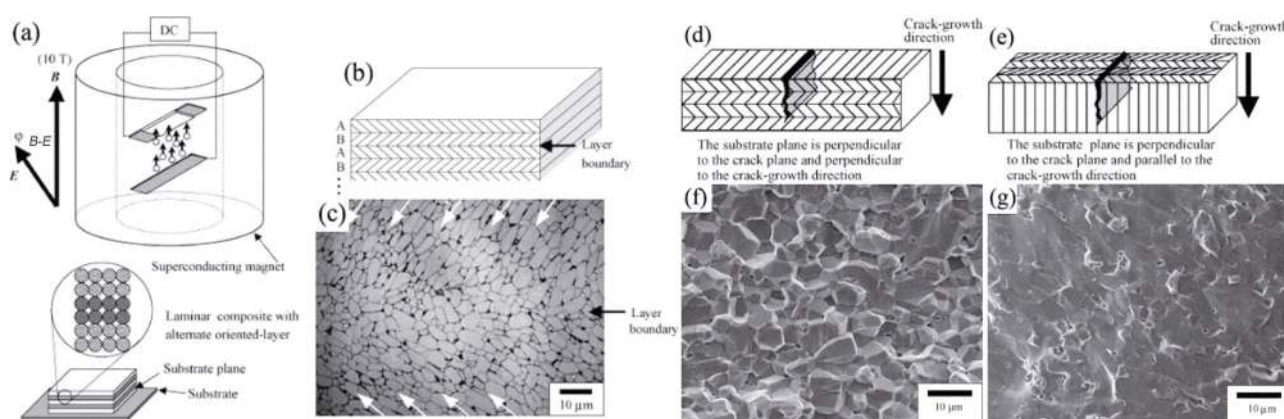


Fig. 23 Preparation and microstructures of multilayered α -Al₂O₃ ceramics by electrophoretic deposition in a magnetic field: (a) schematic diagram of the experimental apparatus; (b, c) microstructure of a multilayered α -Al₂O₃ specimen prepared by alternately changing $\Phi_{B-E} = \pm 45^\circ$; (d, e) schematic diagrams of crack propagation perpendicular and parallel to the substrate, respectively; (f, g) fracture morphologies corresponding to (d) and (e), respectively. Reproduced with permission from Ref. [195], © Elsevier Ltd. 2005.

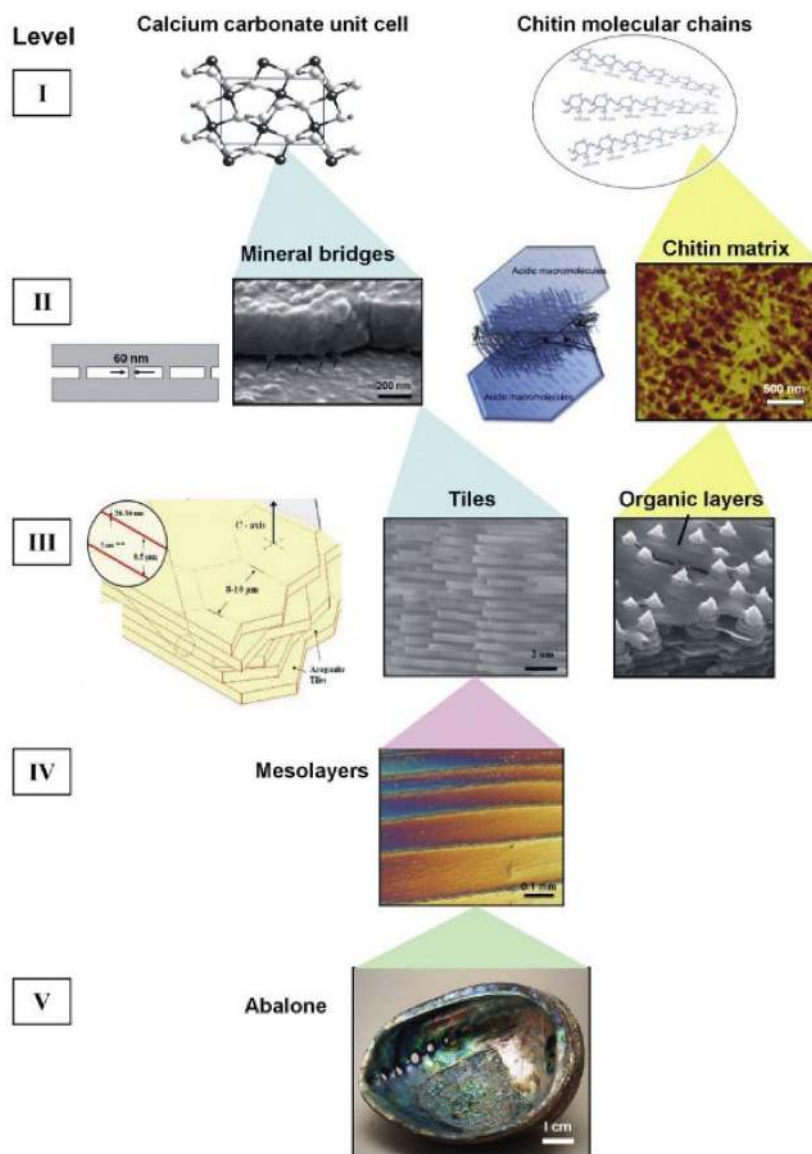


Fig. 24 Five levels of the hierarchical structure of the abalone nacre. (I) Molecular structures of calcium carbonates and chitins. (II) Mineral bridges possessing the sandwich structure. (III) Aragonite tiles and organic layers. (IV) Mesolayers with a thick organic layer separating tile assemblages. (V) The abalone. Reproduced with permission from Ref. [46], © Elsevier Ltd. 2010.

of these platelets, ceramic bonds (bridges) linking these platelets, and a secondary phase with low stiffness to ensure load redistribution, crack deflection, and delamination. This nacre-like $\alpha\text{-Al}_2\text{O}_3$ specimen possesses excellent mechanical properties with no anisotropy. It has a combination of high strength (470 MPa), high toughness (22 MPa·m^{1/2}), and high stiffness (290 GPa). Figure 25(b) shows fracture toughness versus flexural strength of $\alpha\text{-Al}_2\text{O}_3$ -based materials. There are usually conflicts between toughness and strength, because strength is the ability resisting non-recoverable (for example, plastic) deformation, while high toughness is usually related to good plasticity [55,200]. Nacre-like

$\alpha\text{-Al}_2\text{O}_3$ ceramics possess both high strength and toughness due to their hierarchical structure. Besides, they also have specific strength and specific toughness as high as engineering aluminium and magnesium alloys, as shown in Fig. 25(c).

4.2 Si₃N₄ and SiAlON

Silicon nitride (Si₃N₄) belongs to the hexagonal crystal lattice and combines many outstanding properties such as high temperature strength, high thermal conductivity, low thermal expansion coefficient, good resistance to oxidation, excellent creep resistance, and good tribological properties [128,146,201–204]. It has been widely used

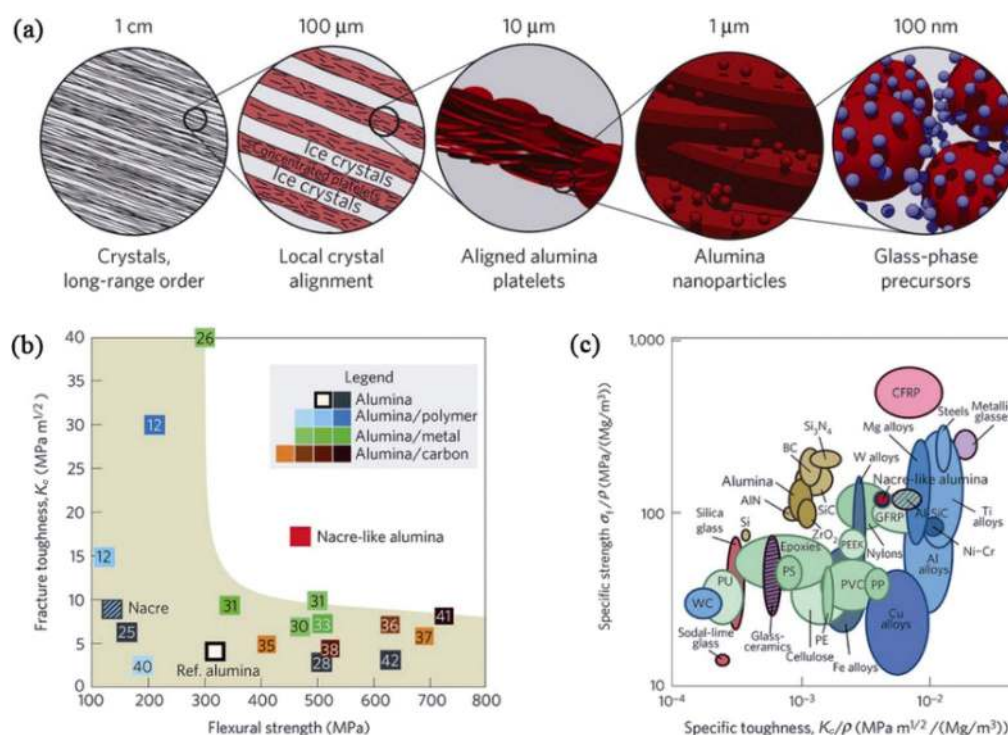


Fig. 25 Microstructure and properties of nacre-like $\alpha\text{-Al}_2\text{O}_3$: (a) long-range ordered structure of nacre-like $\alpha\text{-Al}_2\text{O}_3$; (b) fracture toughness versus flexural strength of $\alpha\text{-Al}_2\text{O}_3$ -based materials (numbers in the figure are the reference numbers in Ref. [55]); (c) ashby diagram of specific strength versus specific toughness of a series of engineering and natural materials. Reproduced with permission from Ref. [55], © Springer Nature 2014.

as high temperature structural materials and has the potential to become the next-generation insulating substrate materials in high-power electronic devices [205,206].

Textured Si_3N_4 ceramics can be prepared using $\alpha\text{-Si}_3\text{N}_4$ powders and $\beta\text{-Si}_3\text{N}_4$ seeds as main raw materials. $\alpha\text{-Si}_3\text{N}_4$ can transfer into $\beta\text{-Si}_3\text{N}_4$ when sintering at high temperature [207]. Some $\beta\text{-Si}_3\text{N}_4$ seeds or rare earth oxides can decrease the activation energy of phase transformation from $\alpha\text{-Si}_3\text{N}_4$ to $\beta\text{-Si}_3\text{N}_4$, promoting phase transformation [208–212]. With some $\beta\text{-Si}_3\text{N}_4$ seeds in $\alpha\text{-Si}_3\text{N}_4$, the nucleation of $\beta\text{-Si}_3\text{N}_4$ grains will take place easily at seed particle. Rod-like Si_3N_4 seeds can grow preferentially along the length direction during sintering and result in a bimodal distribution of grain sizes, that is, a microstructure containing both large elongated grains and fine matrix grains can be obtained. These elongated grains act as the reinforced phase in matrix grains [178,213,214]. In textured Si_3N_4 ceramics containing elongated grains, crack deflection and bridging can be more employed perpendicular to the c -axis orientation, which can significantly improve their mechanical properties [23]. Within a certain range, more $\beta\text{-Si}_3\text{N}_4$ seeds are favorable to preferred orientation

growth and texture formation in Si_3N_4 ceramics [215]. But the preferred orientation growth of Si_3N_4 grains usually happens with the phase transformation from $\alpha\text{-Si}_3\text{N}_4$ to $\beta\text{-Si}_3\text{N}_4$, so high α/β ratio can accelerate the preferred orientation growth of Si_3N_4 grains and improve the mechanical properties [194,216–218].

Si_3N_4 grains can be aligned with the a, b -axis parallel to the magnetic field as $\chi_c < \chi_{a,b}$. So the c -axis will be arbitrarily arranged in the plane perpendicular to the magnetic field [2]. But if the Si_3N_4 suspension mold is rotated in a magnetic field whose direction is parallel to the horizon, Si_3N_4 grains can be aligned parallel to the gravity direction, forming the c -axis oriented texture, as shown in Fig. 26 [133].

When sintered with uniaxial pressure, rod-like Si_3N_4 grains can be rotated to the direction perpendicular to the pressure direction, forming the a, b -axis oriented texture [6]. HF of SC and PSC can result in the a, b -axis oriented texture and the c -axis oriented texture, respectively, as discussed in Section 3.1.1. Figure 27 shows the microstructures and XRD patterns of $\beta\text{-Si}_3\text{N}_4$ specimens possessing the a, b -axis and c -axis oriented texture. For both the two kinds of textured $\beta\text{-Si}_3\text{N}_4$ ceramics, the (002) diffraction intensities on the specimen

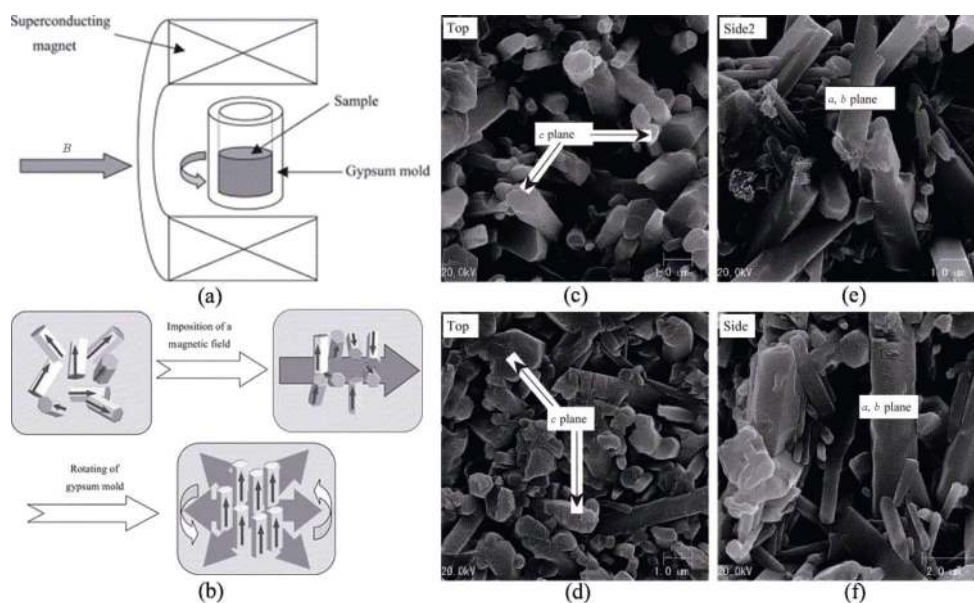


Fig. 26 Preparation and microstructures of textured Si_3N_4 specimens by slip casting in a magnetic field: (a) schematic diagram of the experimental apparatus; (b) effect of the magnetic field and mold rotation on the orientation of rodlike Si_3N_4 grains; (c, e) top and side surfaces of textured Si_3N_4 specimens prepared without mold rotation during slip casting, respectively; (d, f) top and side surfaces of textured Si_3N_4 specimens prepared in the magnetic field with mold rotation during slip casting, respectively. Reproduced with permission from Ref. [133], © Elsevier B.V. 2004.

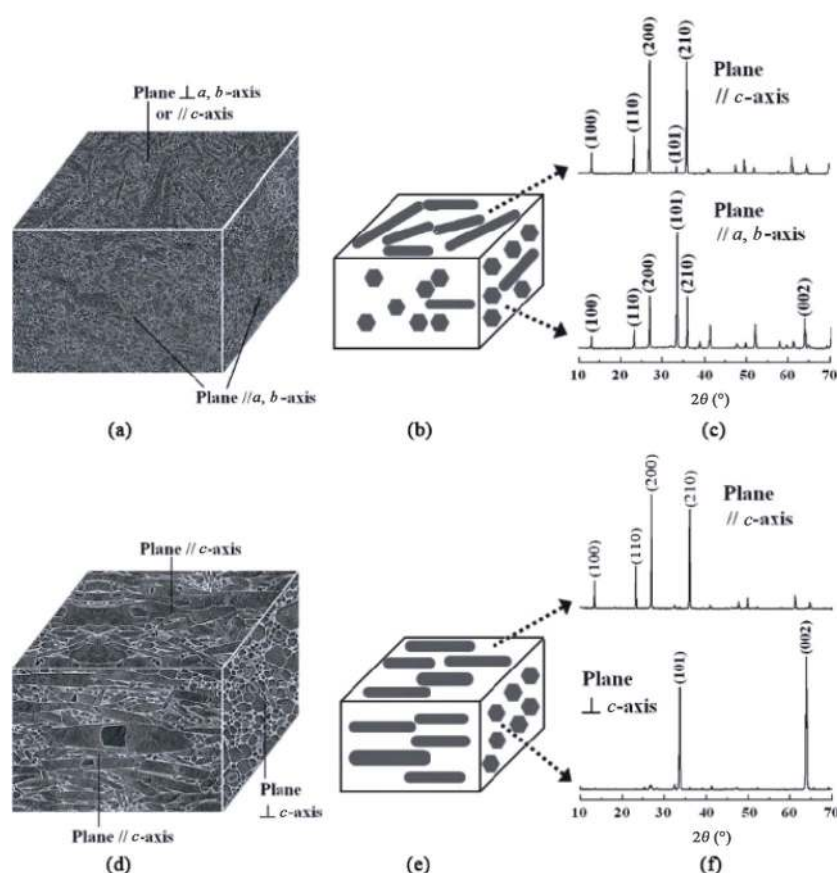


Fig. 27 Microstructures and XRD patterns of the a,b -axis oriented and c -axis oriented $\beta\text{-Si}_3\text{N}_4$ specimens: (a, d) actual microstructures; (b, e) schematic diagrams; (c, f) XRD patterns. Reproduced with permission from Ref. [23], © National Institute for Materials Science 2008.

surfaces parallel to the *a,b*-axis orientation are higher than that parallel to the *c*-axis orientation. But the *c*-axis oriented β - Si_3N_4 specimen shows a much higher (002) diffraction intensity on the specimen surface parallel to the *a,b*-axis orientation than the *a,b*-axis oriented β - Si_3N_4 specimen [23].

Table 6 shows the anisotropic properties of textured Si_3N_4 matrix ceramics. Generally, β - Si_3N_4 seeds with finer size and higher aspect ratio are more favorable to texture formation. Textured Si_3N_4 ceramics with elongated β - Si_3N_4 grains also possess more significantly anisotropic mechanical properties. When the loading direction is perpendicular to the *c*-axis orientation, the grain pull-out mechanism can play a better role strengthening and toughening, resulting in higher mechanical properties [71,213]. However, when the seed amount is enough and the sintering time is very long, the microstructure might be not different from that prepared using near-equiaxed seeds. Because the steric hindrance during prolonged sintering limits the preferred orientation growth. Figure 28 shows the surface morphologies of textured Si_3N_4 ceramics prepared using near-equiaxed and rodlike β - Si_3N_4 seeds [219]. Due to the rotation of

mold in their experiment, Si_3N_4 specimens have the *c*-axis oriented texture. Grain size and morphology are similar for the two specimens. While equiaxed seeds are better to densification and thermal conduction than rod-like seeds.

The thermal conductivity of textured Si_3N_4 ceramics also has strong relationship with grain size. The larger the grain size is, the larger the average free path of phonons is, and the higher thermal conductivity is. The prolonging of holding time, the high temperature annealing after sintering can both promote grain growth [6,220]. The hot-isostatic pressing (HIP) treatment can also lead to further increase of grain size and densification. Watari *et al.* [221] studied the effect of HIP on the thermal conductivity of textured Si_3N_4 ceramics prepared by TGG and HP. Figure 29 shows the surface morphologies and thermal conductivities of textured Si_3N_4 specimens without and with HIP. The HIPed specimen possesses elongated grains with the average diameter of 10 μm and the average length of 200 μm , higher than 1.8 and 10 μm of the specimen without HIP. Thus the HIPed specimen possesses the thermal conductivity of 155 W/(m·K) along the grain alignment

Table 6 Properties of textured Si_3N_4 matrix ceramics

Researchers	Raw materials	Seed morphology	Preparation methods	Relative density/bulk density	Orientation factor	Flexural strength (MPa)	Fracture toughness (MPa·m ^{1/2})	Thermal conductivity (W/(m·K))
Imamura <i>et al.</i> [71]	β - Si_3N_4 + 5 vol% β - Si_3N_4 seeds + 5 mol% Y_2O_3 + 2.5 mol% SiO_2	Diameter = 0.47 μm Aspect ratio = 4.2	TGG & gas pressure sintering	Relative density $\geq 97\%$	$f_0 = 0.35\text{--}0.51$	1396 (<i>// c</i> -axis)	12.4 (<i>// c</i> -axis)	—
		Diameter = 1.29 μm Aspect ratio = 4				724 (\perp <i>c</i> -axis)	7.2 (\perp <i>c</i> -axis)	—
Bae <i>et al.</i> [213]	Si_3N_4 + 3 wt% β - Si_3N_4 seeds + 6 wt% Y_2O_3 + 1 wt% Al_2O_3	Diameter = 1.1 μm Aspect ratio = 11.1	TGG & gas pressure sintering	Relative density $\approx 98\%$	$f_{002} = 0.38$	—	7.1 (<i>// c</i> -axis)	—
		Diameter = 2.8 μm Aspect ratio = 6.7				$f_{002} = 0.3$	6.9 (<i>// c</i> -axis)	5.5 (\perp <i>c</i> -axis)
Pablos <i>et al.</i> [6]	α - Si_3N_4 + 10 wt% Y_2O_3	—	HP (holding time = 1.5 h)	Bulk density = 3.26 g/cm ³	—	—	—	42 (\perp <i>c</i> -axis)
			HP (holding time = 2 h)	Bulk density = 3.26 g/cm ³	—	—	—	82 (<i>// c</i> -axis) 62 (\perp <i>c</i> -axis)
Hirao <i>et al.</i> [220]	α - Si_3N_4 + 5 vol% β - Si_3N_4 seeds + 5 wt% Y_2O_3	Diameter = 1 μm Aspect ratio = 10	TGG & HP & annealing for 2 h	Relative density > 99%	—	—	—	87 (<i>// c</i> -axis) 50 (\perp <i>c</i> -axis)
			TGG & HP & annealing for 66 h	Relative density > 99%	—	—	—	121 (<i>// c</i> -axis) 59 (\perp <i>c</i> -axis)
Watari <i>et al.</i> [221]	α - Si_3N_4 + 5 vol% β - Si_3N_4 seeds + 5 wt% Y_2O_3	Diameter = 1.3 μm Aspect ratio = 4.15	TGG & HP	Bulk density = 3.190 g/cm ³	—	—	—	70 (<i>// c</i> -axis) 30 (\perp <i>c</i> -axis)
			TGG & HP & HIP	Bulk density = 3.205 g/cm ³	—	—	—	155 (<i>// c</i> -axis) 52 (\perp <i>c</i> -axis)
Zhu <i>et al.</i> [219]	α - Si_3N_4 + 5 mol% β - Si_3N_4 seeds + 2 mol% Y_2O_3 + 5 mol% MgSiN_2	Diameter = 0.63 μm Aspect ratio = 1	Magnetic alignment & gas pressure sintering	Relative density = 99.1%	$f_{001} = 0.98$	—	—	176 (<i>// c</i> -axis) 62 (\perp <i>c</i> -axis)
		Diameter = 0.43 μm Aspect ratio = 8.86				Relative density = 97.2%	$f_{001} = 0.97$	—

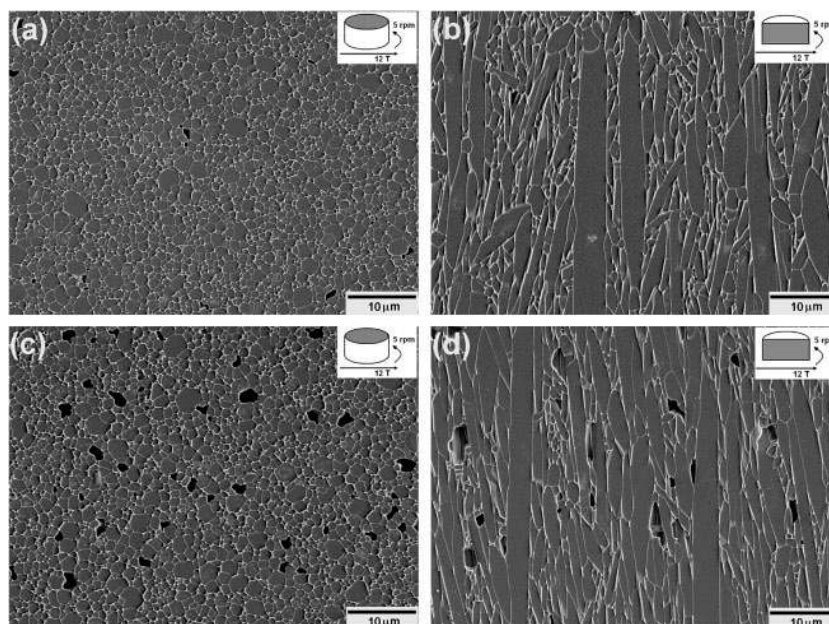


Fig. 28 Polished and plasma-etched surface morphologies of textured Si₃N₄ specimens: (a, b) surfaces perpendicular and parallel to the *c*-axis orientation of the specimen prepared using near-equiaxed β-Si₃N₄ seeds, respectively; (c, d) surfaces perpendicular and parallel to the *c*-axis orientation of the specimen prepared using rodlike β-Si₃N₄ seeds, respectively. Reproduced with permission from Ref. [219], © Elsevier Ltd. 2014.

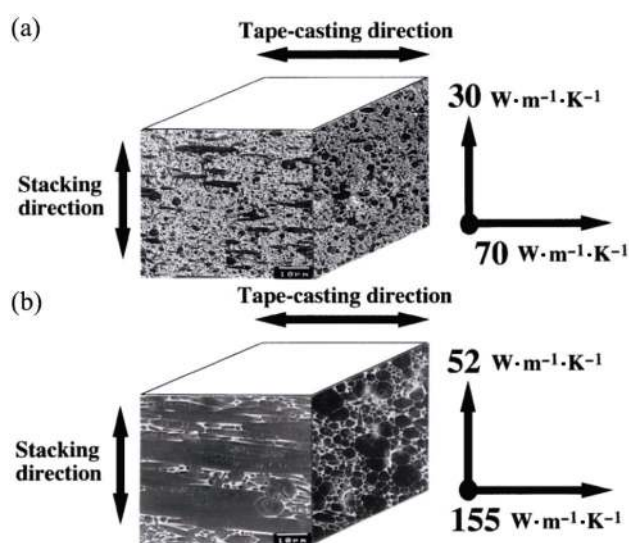


Fig. 29 Plasma-etched surface morphologies and thermal conductivities of textured Si₃N₄ fabricated by: (a) TGG and HP; (b) TGG, HP, and HIP. Reproduced with permission from Ref. [221], © Materials Research Society 1999.

direction, higher than 70 W/(m·K) of that prepared without HIP.

SiAlON is a kind of solid solutions of Si₃N₄ where some Si–N bonds are replaced by Al–O or Al–N bonds. β-SiAlON and α-SiAlON are the solid solutions of β-Si₃N₄ and α-Si₃N₄, respectively [222]. The general formula of α-SiAlON is Me_xSi_{12-(m+n)}Al_{m+n}O_nN_{16-n}, where

m represents the number of Si–N substituted by Al–N, *n* represents the number of Si–N substituted by Al–O, *x* = *m*/*v*, *v* is the chemical valence of Me^{+*v*} cation, and Me is the metal cation compensating for the imbalance of electrovalence to stabilize the structure, usually is Li, Mg, Ca, Y, or some lanthanide [223]. β-SiAlON’s general formula is Si_{6-z}Al_zO_zN_{8-z}, where *z* is the number of N replaced by O and ranges from 0 to 4.2 under the normal pressure [224,225]. α-SiAlON can be prepared by reaction sintering of α-Si₃N₄, Al₂O₃, AlN, and metal oxide powders [226]. β-SiAlON can be prepared by reaction sintering of α-Si₃N₄ or β-Si₃N₄, Al₂O₃, and AlN powders [224,225]. It was found by Muscat *et al.* [179] that there are epitaxial growth of β-SiAlON on β-Si₃N₄ whiskers during reaction sintering, and the nucleation and growth of β-SiAlON also occur between β-Si₃N₄ whiskers simultaneously, forming β-Si₃N₄ whisker-reinforced β-SiAlON composites composed of β-SiAlON matrix and whiskers containing a β-Si₃N₄ core and a β-SiAlON sheath layer.

When the raw material suspension for preparation of SiAlON is placed in a magnetic field, Si₃N₄ grains can be oriented with the *c*-axis perpendicular to the magnetic field. Then after the reaction sintering of the green body, textured SiAlON ceramics can be prepared. Zhu *et al.* [227] prepared Ca-α-SiAlON ceramics using α-Si₃N₄, CaCO₃ (the source of CaO), Al₂O₃, and

AlN as raw materials by slip casting without and with a 12 T magnetic field and subsequent sintering (marked as 0 T specimen and 12 T specimen, respectively). Figure 30 shows the surface morphologies of Ca- α -SiAlON ceramics. The transformation from Si_3N_4 to SiAlON can be completed within 5 min at 1800 °C. The orientation factors I_{210} / I_{102} on the 0 T specimen surfaces perpendicular and parallel to the slip casting direction are 1.02 and 0.92, respectively, with small difference. However, the orientation factors I_{210} / I_{102} on the 12 T specimen surfaces perpendicular and parallel to the magnetic field were 1.51 and 0.72, respectively, indicating the formation of stronger texture. It was revealed by Zhu *et al.* [225] that β - Si_3N_4 is more beneficial to texture formation of β -SiAlON than α - Si_3N_4 . α - Si_3N_4 , β - Si_3N_4 , Al_2O_3 , and AlN powders were used to prepare textured β -SiAlON ceramics by magnetic alignment and sintering. The specimen prepared using β - Si_3N_4 powders containing 3.4 wt% α - Si_3N_4 is higher than 0.97 and even reaches 1 after sintering for 6 h. While that of the specimen prepared using α - Si_3N_4 powders containing 4.6 wt% β - Si_3N_4 is only 0.47 after sintering for 6 h.

Under uniaxial pressure, SiAlON can grow along the c -axis, forming elongated grains perpendicular to

the pressure direction. Normally, for hot working, elongated SiAlON grains are oriented with the c -axis perpendicular to the external pressure, that is, the c -axis can be arbitrarily oriented in the plane perpendicular to the external pressure, forming the a,b -axis oriented texture. But the c -axis oriented texture has been obtained by Carman *et al.* [124] by conducting HF perpendicular to the direction of previous HP. The fracture toughness of α -SiAlON ceramics with the c -axis oriented texture possess more significantly anisotropic fracture toughness (5.7 and 2.9 $\text{MPa}\cdot\text{m}^{1/2}$ perpendicular and parallel to the c -axis orientation, respectively) than that with a,b -axis oriented texture (4.9 and 3.0 $\text{MPa}\cdot\text{m}^{1/2}$ perpendicular and parallel to the c -axis orientation, respectively). The HF after HP can lead to more significant texture and better mechanical properties of SiAlON ceramics due to the grain pull-out toughening mechanism [228].

For textured SiAlON ceramics, the fracture toughness perpendicular to the c -axis orientation is usually higher than that parallel to the c -axis orientation due to deflection and grain pull-out mechanisms. However, if graphene nanoplatelets (GNPs) are introduced during the preparation of SiAlON ceramics, bridging can become the dominant toughening mechanism, and the

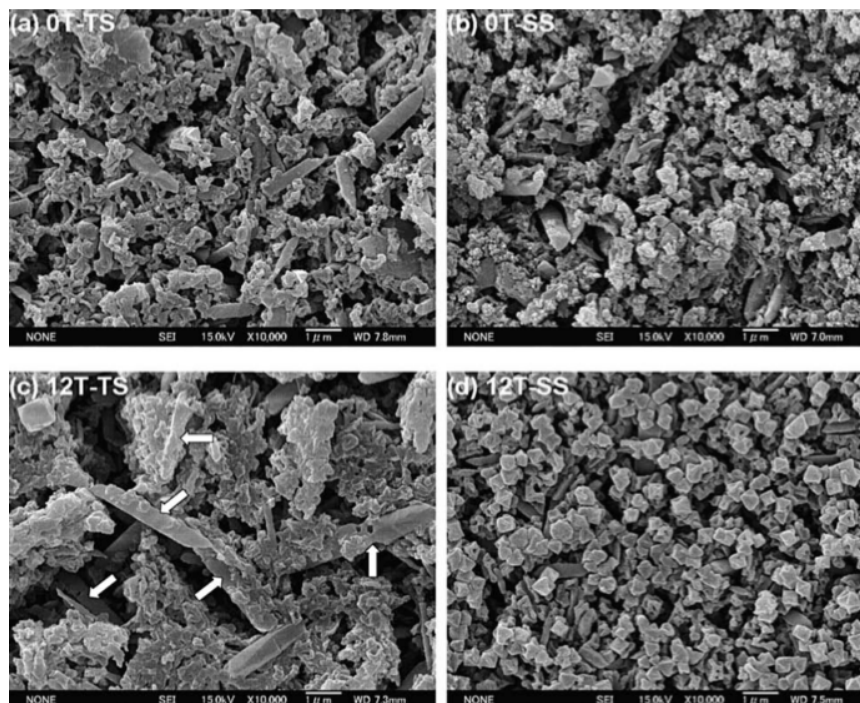


Fig. 30 Polished and molten NaOH-etched surface morphologies of Ca- α -SiAlON ceramics: (a, b) top surface (\perp casting) and side surface (\parallel casting) of 0 T specimen, respectively; (c, d) top surface ($\perp B$) and side surface ($\parallel B$) of 12 T specimen, respectively. White arrows indicate these oriented rodlike grains. Reproduced with permission from Ref. [227], © The Ceramic Society of Japan 2007.

opposite situation may happen. By adding 4 wt% GNPs, the fracture toughness of SiAlON ceramics perpendicular to the *c*-axis orientation decreases from 5.28 to 4.83 MPa·m^{1/2}, while that parallel to the *c*-axis orientation increases from 5.00 to 5.80 MPa·m^{1/2} [229].

4.3 h-BN

Hexagonal boron nitride (h-BN) has a lamellar structure similar to graphite. In each layer, B atoms and N atoms are connected by strong sp² covalent bonds, while different layers are connected by weak van der Waals forces. So h-BN is also called as “white graphite” [230,231]. h-BN has many special properties such as high thermal conductivity, low dielectric coefficient, outstanding thermal shock resistance, etc. [232,233].

Due to the special lamellar structure of h-BN, its properties along different directions are significantly different. Taking its elastic properties as an example, for materials belonging to the hexagonal crystal lattice, there are five independent elastic constants, *c*₁₁, *c*₁₂, *c*₁₃, *c*₃₃, and *c*₄₄ [234]. The five elastic constants of h-BN have been calculated by Xiao *et al.* [235] using the first-principles pseudopotential method, and the five elastic compliance coefficients *s*₁₁, *s*₁₂, *s*₁₃, *s*₃₃, and *s*₄₄ can be calculated according to the relationship between *c*_{*ij*} and *s*_{*ij*}, as shown in Table 7.

The Young’s moduli along [100] and [001] are 1/*s*_{*ij*} and 1/*s*₃₃, respectively [234]. It can be calculated that the Young’s moduli along the *a*-axis and the *c*-axis of h-BN are 837.99 and 33.40 GPa, respectively. The elastic property of h-BN shows obvious anisotropy, so deformation is much harder to occur within the layer than perpendicular to the layer. It is in accordance with the fact that B atoms and N atoms are connected by strong sp² covalent bonds in each layer while different layers are connected by weak van der Waals forces. Besides the elastic property, the thermal conductivity of h-BN also shows obvious anisotropy. The theoretical thermal conductivities parallel and perpendicular to the *c*-axis of bulk h-BN single crystal were calculated to be 4.1 and 537 W/(m·K), respectively [13]. So the

texturing of h-BN matrix ceramics can significantly improve their performances along specific direction and expand their application fields, for example, as heat sinks for semiconductor parts [170].

h-BN is usually textured by HP due to its lamellar structure. It is worth noting that HP will not definitely lead to texture microstructures where the *c*-axis of h-BN is parallel to the external pressure. Sometimes, when the h-BN raw powders have a low degree of order and a broad size distribution, no or little sintering additive is used, the external pressure is moderate, and the temperature is high enough, a completely different texture can be produced, where the *c*-axis can be oriented perpendicular to the pressure direction. This is because that the uniaxial pressure has two effects on h-BN grains during sintering. One is called “grow effect”, promoting the contact of h-BN grains and the plate-like grain growth along the pressure direction. The other is called “rotate effect”, making plate-like h-BN grains rotated to be perpendicular to the pressure direction. The texture microstructures of sintered h-BN specimens are determined by the leading effect [170,236]. When the temperature is high enough and the pressure is not very high, the “grow effect” will be dominant, because high temperature can accelerate atomic diffusion, which is beneficial to the grain growth. In the meantime, the moderate pressure cannot rotate grains dramatically, so the *c*-axis tends to be perpendicular to the pressure direction. When the temperature is not very high and the pressure is high enough, the “rotate effect” will be dominant, leading to a microstructure where the *c*-axis tends to be parallel to the pressure direction. The two different texture microstructures can lead to different anisotropy. However, h-BN ceramics HPed with no or little sintering additive can hardly possess dramatic texture [1,170].

The mechanical and thermal properties of textured h-BN matrix ceramics are displayed in Table 8. Fracture mechanisms under different loading situations of textured h-BN matrix ceramics with lamellar structure are shown in Figs. 31(a), 31(c), and 31(e). Figures 31(b), 31(d), and 31(f) show the corresponding fracture morphologies of textured h-BN–mullite composites [4]. The mechanical properties are the worst when the crack propagates between h-BN layers, corresponding to D1 loading situation shown in Fig. 31(a). When the external force is perpendicular to h-BN layers, the crack needs to go through a longer path to make it fracture, corresponding to D2 in Fig. 31(c). Another

Table 7 Elastic constants and compliance coefficients of h-BN at room temperature, *P* = 0 [235]

<i>c</i> _{<i>ij</i>} (GPa)	<i>c</i> ₁₁	<i>c</i> ₁₂	<i>c</i> ₁₃	<i>c</i> ₃₃	<i>c</i> ₄₄
	880.59	193.39	2.7	33.41	5.22
<i>s</i> _{<i>ij</i>} (TPa ⁻¹)	<i>s</i> ₁₁	<i>s</i> ₁₂	<i>s</i> ₁₃	<i>s</i> ₃₃	<i>s</i> ₄₄
	1.193	-0.262	-0.075	29.943	191.571

Table 8 Properties of textured h-BN matrix ceramics

Researchers	Raw materials	Sintering additives	Preparation method	Relative density/ bulk density	Orientation factor	Elastic modulus (GPa)	Flexural strength (MPa)	Fracture toughness (MPa·m ^{1/2})	Thermal conductivity (W/(m·K))
Xue <i>et al.</i> [170]	Irregular shaped h-BN	—	HP	Relative density = 93.5%	$IOP = 27.2$	—	55.2 (D1) 43.1 (D2)	—	67 (<i>// c-axis</i>) 35 ($\perp c-axis$)
				HP (10 MPa)	Bulk density = 2.00 g/cm ³	$IOP = -178$	3.68 (D1) 64.64 (D2) 70.44 (D3)	23.68 (D1) 74.49 (D2) 43.90 (D3)	0.41 (D1) 1.20 (D2) 1.22 (D3)
Duan <i>et al.</i> [4,236]	Plate-like h-BN	20 wt% mullite	HP (20 MPa)	Bulk density = 2.09 g/cm ³	$IOP = -350$	5.34 (D1) 69.78 (D2) 84.02 (D3)	27.61 (D1) 93.26 (D2) 81.58 (D3)	0.42 (D1) 1.49 (D2) 2.00 (D3)	—
				HP (30 MPa)	Bulk density = 2.17 g/cm ³	$IOP = -2160$	5.82 (D1) 90.32 (D2) 107.77 (D3)	28.93 (D1) 100.22 (D2) 104.95 (D3)	0.45 (D1) 2.08 (D2) 2.36 (D3)
Zhang <i>et al.</i> [233]	Plate-like h-BN	20 wt% 3Y ₂ O ₃ -5Al ₂ O ₃	HP	Relative density = 88.7%	$IOP = -530$	8.56 (D1) 66.86 (D2) 82.76 (D3)	16.46 (D1) 73.07 (D2) 77.92 (D3)	0.36 (D1) 1.87 (D2) 2.10 (D3)	17.62 (<i>// c-axis</i>) 154.62 ($\perp c-axis$)
		20 wt% 3Y ₂ O ₃ -5Al ₂ O ₃ -4MgO		Relative density = 89.6%	$IOP = -976$	6.91 (D1) 83.15 (D2) 88.04 (D3)	17.53 (D1) 81.78 (D2) 84.03 (D3)	0.45 (D1) 2.00 (D2) 2.36 (D3)	22.74 (<i>// c-axis</i>) 137.01 ($\perp c-axis$)
Ni <i>et al.</i> [79]	Irregularly shaped h-BN	1.98 wt% SiO ₂ + 0.36 wt% Al ₂ O ₃ + 2.66 wt% Y ₂ O ₃	SPS	Relative density = 92.0%	$MRD = 7.6$	—	—	—	29.8 (<i>// c-axis</i>) 35.5 ($\perp c-axis$)
	Plate-like h-BN			Relative density = 88.9%	$MRD = 15.4$	—	—	—	15.3 (<i>// c-axis</i>) 23.7 ($\perp c-axis$)
Kusunose and Sekino [127]	Plate-like h-BN	15 vol% MgO	HP	—	—	—	—	—	14 (<i>// c-axis</i>) 113 ($\perp c-axis$)
		15 vol% Yb ₂ O ₃ -MgO		—	—	—	—	—	14 (<i>// c-axis</i>) 212 ($\perp c-axis$)
		5 vol% Yb ₂ O ₃ -MgO		—	—	—	—	—	12 (<i>// c-axis</i>) 173 ($\perp c-axis$)

loading situation is D3 in Fig. 31(e), that is, although the loading force is parallel to the layers, the initial crack is tortuous and the crack path is similar to that of D2. To generate initial crack for D2, only chemical bonds in one h-BN layer need to be broken. When it comes to D3, not only chemical bonds but also van der Waals forces are broken. So the flexural strength of D3 is a little higher than that of D2.

As discussed in Section 3.2.1, the composite additives of a rare-earth oxide and an alkaline-earth oxide are better to the texture formation and directional thermal conduction performance of HPed h-BN matrix ceramics than the simple additive of an alkaline-earth oxide. Our previous study shows that Y₃Al₅O₁₂ liquid phase formed from Y₂O₃ and Al₂O₃ sintering additives has good wettability to h-BN grains [233]. The fracture morphologies and polished surfaces of HPed h-BN matrix ceramics using 15 vol% MgO and 15 vol% Yb₂O₃-MgO as additives are shown in Fig. 32 [127]. Yb₂O₃-MgO additives are distributed in h-BN matrix more homogeneously. The specimen sintered with 15 vol% Yb₂O₃-MgO possesses larger grain size and more significant texture and better directional thermal

conduction performance. Yb₂O₃-MgO exhibits better wettability to h-BN grains than the single component MgO additive, thus promoting preferred orientation growth via solution reprecipitation and improving the thermal conductivity.

4.4 MB₂ matrix ultra-high temperature ceramics

Ultra-high temperature ceramics (UHTCs) are a class of ceramics that have melting point above 3000 °C and can be used at temperatures above 1650 °C continually. Almost all UHTCs are borides, carbides, and nitrides of early transition metals such as Zr, Hf, Ti, and Ta [184,237]. UHTCs have high strength and hardness, good thermal shock resistance, and good chemical stability [238–240]. Their good properties at extreme high temperature allow them to be used in aerospace sharp-shaped hot-structures, leading edge parts on re-entry vehicles, etc. [183,241,242].

Among the family of UHTCs, transition metal diboride (MB₂) belongs to the hexagonal crystal lattice and is easy to be textured under the external magnetic field or uniaxial pressure. MB₂ matrix ceramics can be prepared by both non-reactive sintering and reactive

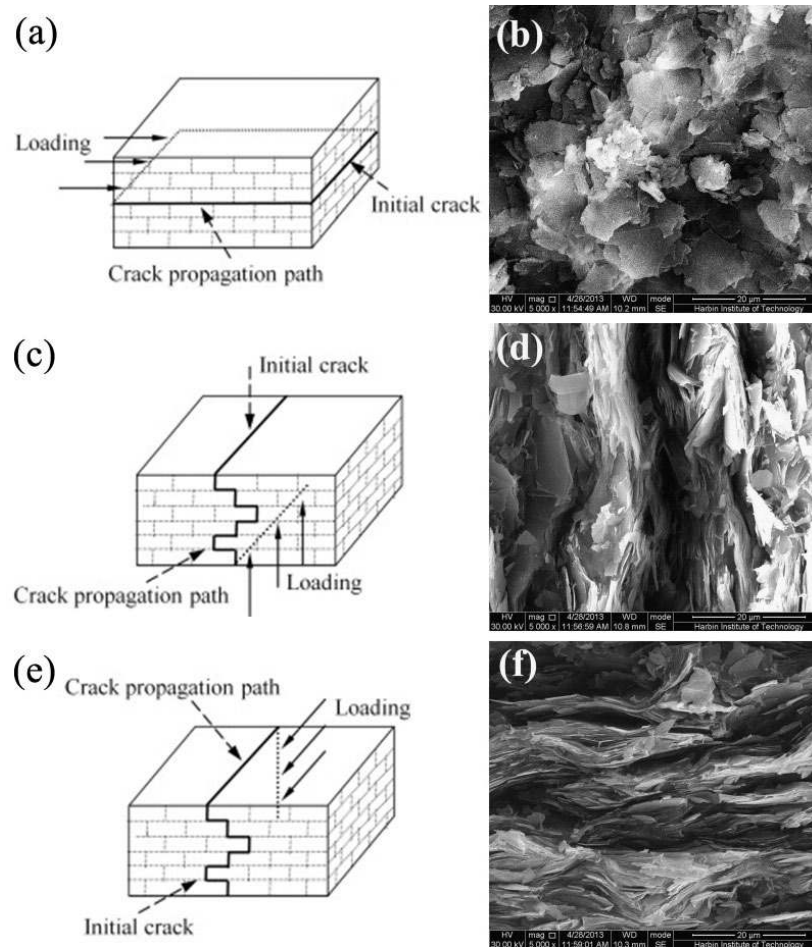


Fig. 31 Fracture mechanisms and fracture morphologies of textured h-BN-mullite composites with lamellar structure under different loading situations: (a, b) D1; (c, d) D2; (e, f) D3. Reproduced with permission from Ref. [4], © Elsevier B.V. 2014.

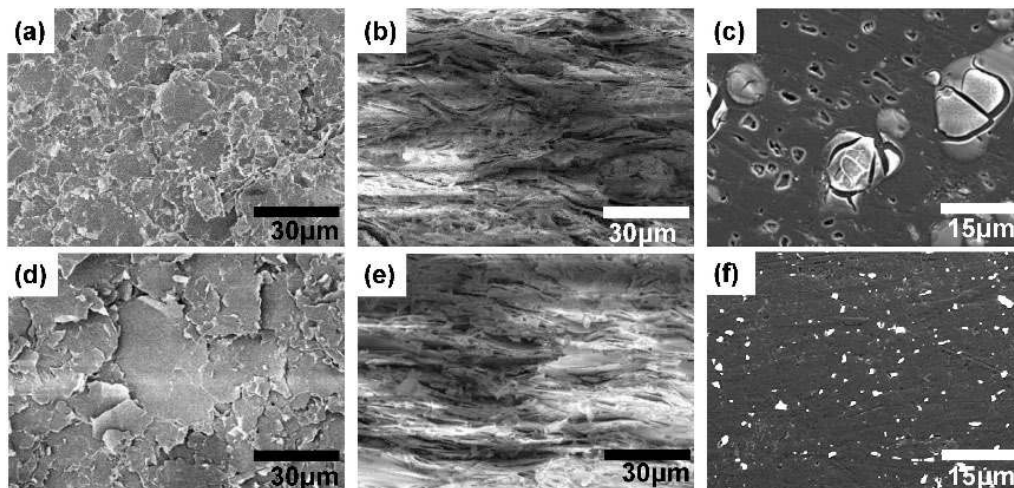


Fig. 32 Fracture morphologies perpendicular (a, d) and parallel (b, e) to the pressure direction and (c, f) polished surfaces perpendicular to the pressure direction of HPed h-BN matrix ceramics with different additives: (a–c) 15 vol% MgO; (d–f) 15 vol% Yb₂O₃–MgO. Reproduced with permission from Ref. [127], © Acta Materialia Inc. 2016.

sintering which combines synthesis and densification into one step using elemental powders [239,243,244]. The high temperature oxidation resistance of pure MB₂

ceramics is limited due to the evaporation of B₂O₃ above 1200 °C, which makes oxygen diffuse more easily into MB₂ substrate. But their oxidation resistance

can be improved when combined with SiC, which can provide an outer SiO₂-rich glassy layer acting as a barrier to diffusion of oxygen to the MB₂ substrate [183,243, 245]. Among MB₂ matrix ceramics, ZrB₂ and HfB₂ based ceramics are the most two promising candidates for extreme environmental applications. While the application of TiB₂ is limited to armor and cutting tools [246–248].

The preferred orientation growth of MB₂ grains are strongly depended on liquid phase environment during sintering [245,249,250]. As UHTCs, MB₂ matrix ceramics are demanded to have high melting temperature, and there is usually no low melting point substance in the sintered specimens. So reactive sintering became an effective method to prepare MB₂ matrix ceramics with strong morphological texture. During reactive sintering, these single elements can form low melting point liquid phases, which are favorable to preferred orientation growth and grain rotation under external pressure.

Wu *et al.* [251] prepared highly textured ZrB₂–5 vol% B₄C composites by slip casting in a magnetic field followed by SPS. The XRD patterns and surface morphologies perpendicular (TS) and parallel (SS) to the magnetic field direction are presented in Fig. 33. The main diffraction peaks on TS and SS are (001) and (100), respectively. The *c*-axis of ZrB₂ is oriented parallel to the magnetic field direction. The Lotgering factor f_{00l} is as high as 0.92. But there is no obvious difference between surface morphologies of SS and TS. The grain sizes for the TS and SS are 2.5 and 2.4 μm, respectively, indicating there is no preferred orientation growth of ZrB₂ grains during sintering. Liu *et al.* [250] prepared textured and platelet-reinforced ZrB₂–20 vol% MoSi₂ composites by reactive hot pressing (RHP) and subsequent hot forging (HF) with simple compression using powders of elemental Zr, B, Mo, and Si. Figure 34 shows the XRD, TEM, and SEM analysis of the

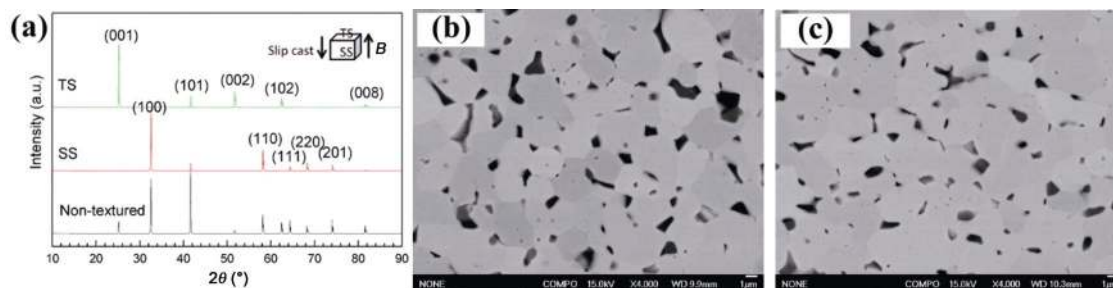


Fig. 33 XRD patterns and surface morphologies of textured ZrB₂–5 vol% B₄C composites prepared by magnetic alignment and SPS: (a) XRD patterns of TS and SS; (b, c) surface morphologies of TS and SS, respectively. Reproduced with permission from Ref. [251], © National Institute for Materials Science 2014.

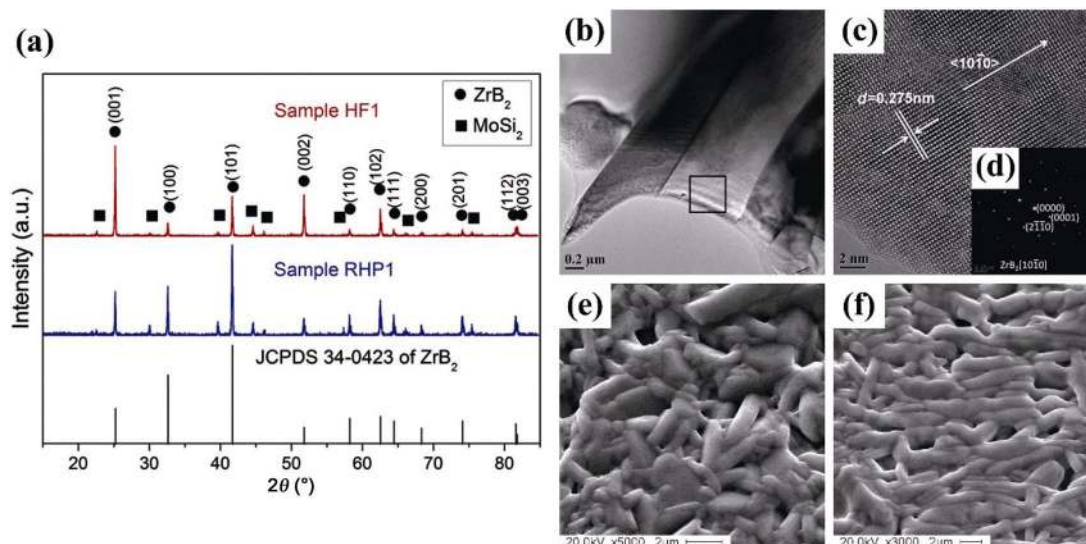


Fig. 34 XRD, TEM, and SEM analysis of textured ZrB₂–20 vol% MoSi₂ composites prepared by RHP and HF: (a) XRD patterns of specimen surfaces perpendicular to the pressure direction; (b, c, d) TEM image, HRTEM image, and selected-area electron diffraction pattern of ZrB₂ platelet grains, respectively; (e, f) SEM images of polished surface parallel to the pressure direction for sample RHP1 and HF1, respectively. Reproduced with permission from Ref. [250], © Acta Materialia Inc. 2011.

as-RHPed specimen before HF (RHP1) and the specimen after HF (HF1). The *c*-axis of ZrB₂ is oriented parallel to the pressure direction, and the Lotgering factors *f*₀₀₁ of the RHP1 and HF1 specimens are 0.33 and 0.59, respectively, indicating HF after RHP further enhanced the crystallographic texture. The TEM analysis demonstrated the preferred orientation growth of platelet ZrB₂ grains along the *a,b*-axis, whose morphologies are shown in the SEM images. After HF, the platelet ZrB₂ grains further grew from 0.57 μm in thickness and 2.28 μm in diameter to 0.95 μm in thickness and 5.71 μm in diameter. The anisotropic grain growth is probably related to the Zr–Mo/Si–Mo liquid phase. The more elongated grains after HF are favorable to grain rotation under applied pressure, resulting in more significant morphological and crystallographic texture compared with the as-RHPed specimen before HF.

Table 9 lists anisotropic properties of textured MB₂ matrix ceramics. The mechanical properties of textured MB₂ matrix ceramics are strongly related to grain orientation, grain shape, and slip plane. It was demonstrated the slip occurred along <1000> direction on prismatic {10 $\bar{1}$ 0} planes at room temperature but on the basal {0001} plane at high temperature in the ZrB₂ crystal [252]. For textured ZrB₂–5 vol% B₄C composites with equiaxed grains prepared by Wu *et al.* [251], there is more plastic deformation to increase the required strength for fracture when the loading direction is perpendicular to the *c*-axis orientation than parallel to the *c*-axis orientation at low temperature. However, the

opposite situation happens at high temperatures. The higher strength at 1600 °C than at room temperature is also related to the more plastic deformation at high temperature and besides, the suppression of crack extension along the grain boundaries due to the strong bonding of clean ZrB₂/ZrB₂ interfaces. However, for textured ZrB₂ matrix ceramics composed of elongated grains or platelet grains, the influence of grain shape on the anisotropy of mechanical properties is more remarkable than that of slip plane [249,250].

HfB₂ matrix ceramics have better oxidation resistance than ZrB₂ matrix ceramics. Ni *et al.* [253,254] studied the oxidation resistance of textured ZrB₂–20 vol% SiC composites (ZS20) and textured HfB₂–20 vol% SiC composites (HS20) prepared by magnetic alignment and SPS with the same process parameters. After holding at 1600 °C for 10 min in stagnant air, there are SiO₂-rich glassy layers of 40 and 15 μm on the surfaces perpendicular and parallel to the magnetic field direction for ZS20, respectively, but there is no obvious oxidation on both surfaces perpendicular and parallel to the magnetic field direction for HS20. Figure 35 shows the cross-section images of HS20 after oxidation at 1600 °C for 10 h in stagnant air. There are SiO₂-rich glassy layers of 60 and 20 μm on the surfaces perpendicular and parallel to the magnetic field direction, respectively. There is no oxygen in HfB₂–SiC matrix on the surface perpendicular to the magnetic field direction, but there is a HfO₂–SiO₂ layer of 60 μm on the surface parallel to the magnetic field

Table 9 Properties of textured MB2 matrix ceramics

Researchers	Compositions	Preparation methods	Relative density/bulk density	Lotgering factor <i>f</i> ₀₀₁	Vickers hardness (GPa)	Flexural strength (MPa)	Fracture toughness (MPa·m ^{1/2})
Ni <i>et al.</i> [254]	ZrB ₂ –20 vol% SiC	Magnetic alignment & SPS	Relative density = 96.2%	0.94	16.9 (<i>// c</i> -axis) 14.7 (\perp <i>c</i> -axis)	—	3.94 (<i>// c</i> -axis) 3.86 (\perp <i>c</i> -axis)
Ni <i>et al.</i> [253]	HfB ₂ –20 vol% SiC	Magnetic alignment & SPS	Relative density = 98.3%	0.91	21.1 (<i>// c</i> -axis) 19.3 (\perp <i>c</i> -axis)	—	5.9 (<i>// c</i> -axis) 5.5 (\perp <i>c</i> -axis)
	HfB ₂ –5 vol% SiC		Relative density = 97.6%	0.91	20.6 (<i>// c</i> -axis) 18.9 (\perp <i>c</i> -axis)	—	5.8 (<i>// c</i> -axis) 5.5 (\perp <i>c</i> -axis)
Ran <i>et al.</i> [245]	ZrB ₂ –20 vol% SiC	Reactive SPS	Bulk density = 5.62 g/cm ³	0.17	21.3 (<i>// c</i> -axis) 19.7 (\perp <i>c</i> -axis)	937	3.9 (<i>// c</i> -axis) 4.7 (\perp <i>c</i> -axis)
Wu <i>et al.</i> [249]	ZrB ₂ –20 vol% MoSi ₂	Reactive HP	Relative density = 99.2%	—	15	456.9	4.9
Liu <i>et al.</i> [250]	ZrB ₂ –20 vol% MoSi ₂	Reactive HP	—	0.33	—	572 (<i>// c</i> -axis) 499 (\perp <i>c</i> -axis)	—
		Reactive HP & HF	Relative density > 98%	0.59	—	871 (<i>// c</i> -axis) 723 (\perp <i>c</i> -axis)	—
Wu <i>et al.</i> [251]	ZrB ₂ –5 vol% B ₄ C	Magnetic alignment & SPS	Bulk density = 5.78 g/cm ³	0.92	19.1 (<i>// c</i> -axis) 17.5 (\perp <i>c</i> -axis)	574 (<i>// c</i> -axis, RT) 723 (\perp <i>c</i> -axis, RT) 810 (<i>// c</i> -axis, 1600 °C) 650 (\perp <i>c</i> -axis (1600 °C)	—

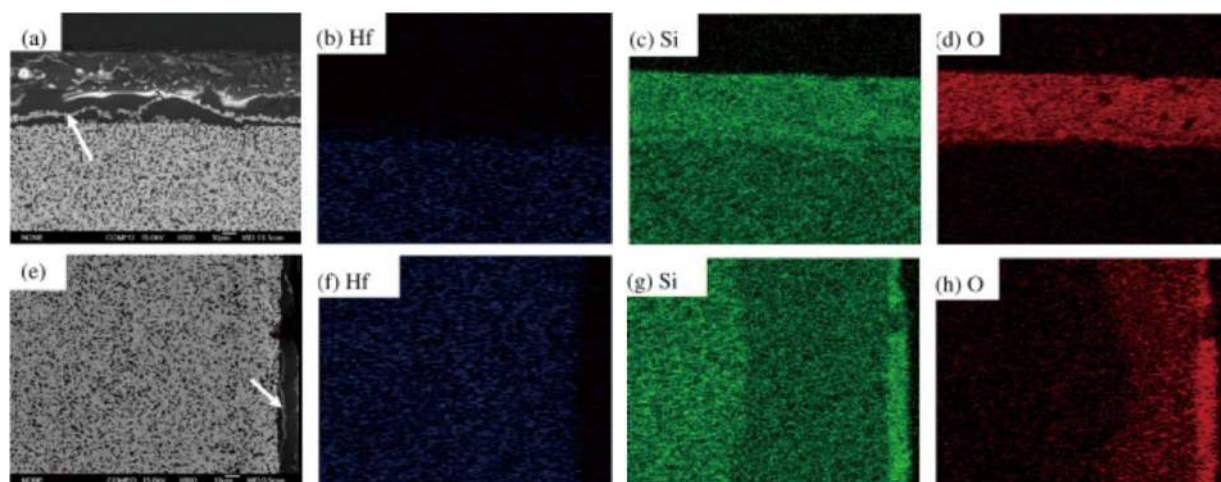


Fig. 35 Cross-section images and EDS elemental maps of different surfaces for HS20 after oxidation at 1600 °C for 10 h in stagnant air: (a–d) the surface perpendicular to the magnetic field direction; (e–h) the surface parallel to the magnetic field direction. Reproduced with permission from Ref. [253], © Acta Materialia Inc. 2009.

direction, indicating the thicker SiO₂-rich glassy layer protect the matrix from oxidation better on the surface perpendicular to the magnetic field direction. The results are the same for ZS20. The anisotropic oxidation resistance of textured MB₂ matrix ceramics might be related to the anisotropic mass transmission of oxygen.

4.5 MAX phases

MAX phases are the family of carbide and nitride ternary ceramics, which belong to the hexagonal crystal lattice and possess lamellar structure. Their general formula is M_{n+1}AX_n, where $n = 1-3$, M is a transition metal, A is an A group element, and X is C or N. Their spacial structure can be described as M_{n+1}X_n layers and A layers alternatively stacking along the *c*-axis. Chemical bonds in MAX phases are a combination of metallic, covalent, and ionic bonds. M and X are combined by strong covalent bonds or ionic bonds, while M and A are combined by weak covalent bonds or metallic bonds. MAX phases can be divided into 211 phases, 312 phases, and 413 phases according to the “*n*” value. For M_{n+1}AX_n, there are $n+1$ M layers between two A layers [255–257]. Besides, some other phases have been discovered in the MAX family, such as 615 phases (Ta₆AlC₅) and 523 phases (Ti₅Al₂C₃) [258]. MAX phases have been widely studied due to their excellent properties combining that of metals and ceramics, such as ductile and machinable, strong and light, good electrical and thermal conductivities, good resistance to oxidation and thermal shock, good wear resistance, etc. [180,185–189].

Due to the weak combination between M_{n+1}X_n layers

and A layers, A element tends to diffuse more easily than M and X and can be oxidized when exposed to oxygen at high temperature [259]. Those existing cracks can be filled by the volume expansion resulting from the oxidation of the A element at high temperature. So MAX phases have been widely used as self-healing materials [260,261].

MAX phases can be synthesized by solid reaction of powders of M, A, and X elements. The synthetic MAX phases are ball milled to form fine plate-like powders, and then textured MAX phase ceramics are prepared by hot working or magnetic alignment [262–267]. Some MAX phases can be oriented with the *c*-axis parallel to the magnetic field direction but some may be opposite. That depends on their anisotropic magnetic susceptibility. For example, Ti₃SiC₂ grains tend to be oriented with the *c*-axis perpendicular to the magnetic field direction, while Nb₄AlC₃ grains tend to be oriented with the *c*-axis parallel to the magnetic field direction [131,264].

Textured MAX phase ceramics prepared using fine plate-like powders after ball milling usually show less damage tolerance than untextured MAX phase ceramics prepared using coarse powders due to the difference between grain sizes. Figure 36 shows the Vickers indentation of the untextured coarse-grained Cr₂AlC specimen and textured fine-grained Cr₂AlC specimen [123]. The textured Cr₂AlC specimen is more brittle than the untextured one and cracks appear at the indentation corners. On the surface parallel to the *c*-axis orientation, it is more easily for the crack to propagate perpendicular to the *c*-axis orientation due to

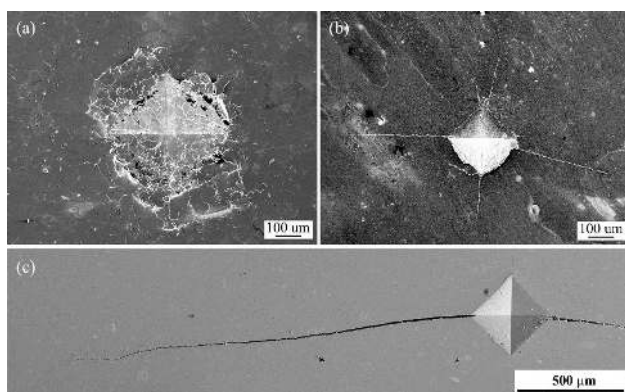


Fig. 36 Morphologies of Vickers indentation of untextured and textured Cr₂AlC specimens: (a) untextured specimen; (b, c) textured specimen surfaces perpendicular and parallel to the sintering pressure, respectively (the double arrow indicates the pressure direction). Reproduced with permission from Ref. [123], © Elsevier Ltd. 2014.

the lamellar texture.

Table 10 lists the anisotropic properties of some typical textured MAX phase ceramics. Due to the lamellar structure, the hardness of MAX phase ceramics along the *c*-axis orientation is normally lower than that perpendicular to the *c*-axis orientation, which has been proved by Mizuno *et al.* [263]. The textured Ti₃AlC₂ specimen prepared by SPS has a Lotgering factor f_{00l} of 0.69, and the Vickers hardness under a load of 10 N parallel and perpendicular to the *c*-axis orientation is 5.9 and 7.0 GPa, respectively. But when the load is high enough, an opposite situation may happen. Zhang *et al.* [266] prepared Ti₃AlC₂ ceramics by magnetic alignment and subsequent SPS. When the load reaches 100 N, the Vickers hardness along the

c-axis orientation is 7.4 GPa, higher than 5.8 GPa perpendicular to the *c*-axis orientation. This phenomenon is attributed to the irreversible plastic deformation along the *c*-axis.

If oxygen is introduced during ball milling of MAX phases, oxides of A element can be formed *in situ* during sintering. These oxide particles homogeneously distributed in the MAX matrix play a significant role of deflecting and blunting the cracks, which are favorable to the improvement of mechanical properties. Figure 37 shows the crack propagation of textured Ti₃AlC₂ specimen [266]. The crack deflection and grain pull-out on the surface parallel to the *c*-axis orientation and the crack deflection and grain bridging on the surface perpendicular to the *c*-axis orientation are the main energy dispersive modes, which contribute to high flexural strength and fracture toughness on both surfaces.

The anisotropy of electrical conductivities of different MAX phase ceramics may be different. Textured Ti₃AlC₂ ceramics possess higher thermal conductivity perpendicular to the *c*-axis orientation and higher electrical conductivity parallel to the *c*-axis orientation [266]. While textured Nb₄AlC₃ ceramics possess both higher thermal conductivity and higher electrical conductivity perpendicular to the *c*-axis orientation [268]. For MAX phases, both phonons and electrons contribute to thermal conduction, but only electrons contribute to electrical conduction. Due to the lamellar structure of MAX phases, phonons are less scattered along the *a,b*-axis orientation, causing higher thermal conductivity. For Nb₄AlC₃, Nb *d* electrons mainly contribute to the density of states (DOS) around the

Table 10 Properties of textured MAX phases

Researchers	Composition	Preparation methods	Lotgering factor f_{00l}	Vickers hardness (GPa)	Flexural strength (MPa)	Fracture toughness (MPa·m ^{1/2})	Thermal conductivity (W/(m·K))	Electrical conductivity (10 ⁶ Ω ⁻¹ ·m ⁻¹)
Zhang <i>et al.</i> [266]	Ti ₃ AlC ₂	Magnetic alignment & SPS	—	8.1 (<i>// c</i> -axis, 1 N) 9.1 (\perp <i>c</i> -axis, 1 N) 7.4 (<i>// c</i> -axis, 100 N) 5.8 (\perp <i>c</i> -axis, 100 N)	1261 (<i>// c</i> -axis) 1005 (\perp <i>c</i> -axis)	13.1 (<i>// c</i> -axis) 14.6 (\perp <i>c</i> -axis)	14.6 (<i>// c</i> -axis) 25.3 (\perp <i>c</i> -axis)	1.01 (<i>// c</i> -axis) 0.83 (\perp <i>c</i> -axis)
Mizuno <i>et al.</i> [263]	Ti ₃ AlC ₂	SPS	0.69	5.9 (<i>// c</i> -axis, 10 N) 7.0 (\perp <i>c</i> -axis, 10 N)	—	—	—	—
Sato <i>et al.</i> [265]	Ti ₃ SiC ₂	Magnetic alignment & pressureless sintering	0.97	—	623	5.9	—	3.23
Hu <i>et al.</i> [131]	Ti ₃ SiC ₂	SPS	0.95	8.70 (<i>// c</i> -axis, 9.8 N) 7.31 (\perp <i>c</i> -axis, 9.8 N)	—	—	—	—
Hu <i>et al.</i> [268]	Nb ₄ AlC ₃	Magnetic alignment & SPS	~1	10.4 (<i>// c</i> -axis, 0.245 N) 12.4 (\perp <i>c</i> -axis, 0.245 N) 7.0 (<i>// c</i> -axis, 49 N) 4.9 (\perp <i>c</i> -axis, 49 N)	881 (<i>// c</i> -axis) 789 (\perp <i>c</i> -axis)	14.1 (<i>// c</i> -axis) 9.3 (\perp <i>c</i> -axis)	14.1 (<i>// c</i> -axis) 21.1 (\perp <i>c</i> -axis)	0.49 (<i>// c</i> -axis) 0.81 (\perp <i>c</i> -axis)

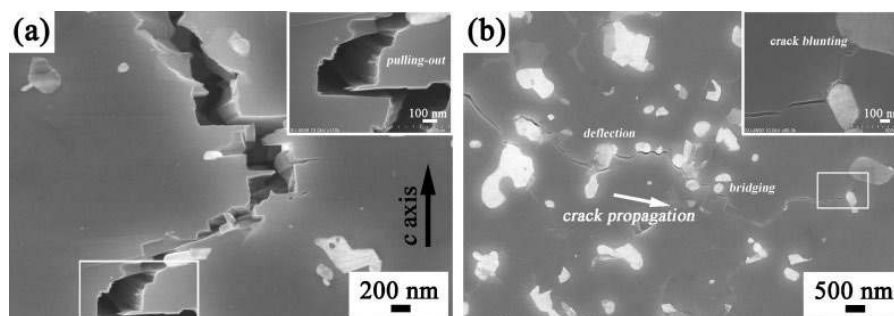


Fig. 37 Crack propagation on different polished surfaces of textured Ti_3AlC_2 ceramic: (a) parallel to the c -axis orientation; (b) perpendicular to the c -axis orientation. The inset micrographs are the magnified region in the figures. Reproduced with permission from Ref. [266], © Elsevier Ltd. 2014.

Fermi level. So textured Nb_4AlC_3 ceramics possess higher electrical conductivity along the a,b -axis orientation [269]. For textured Ti_3AlC_2 , the metallic bonds between Ti and Al mainly contribute to the electrical conductivity. So textured Ti_3AlC_2 ceramics possess higher electrical conductivity along the c -axis orientation, where Ti and Al planes are stacked [270].

4.6 Other ceramics

Besides these typical textured structural ceramics discussed in Sections 3.1–3.4, there are also many other structural ceramics can be textured to improve their performances, such as silicon carbide (SiC), yttria partially stabilized tetragonal zirconia (Y-TZP), boron carbide (B_4C), etc. SiC is one important high temperature structural material due to its excellent mechanical, thermal, and chemical properties [271,272]. Y-TZP is frequently used as thermal barrier coatings [273]. B_4C is one kind of important hard materials [274].

α -SiC belongs to the hexagonal crystal lattice and usually presents plate-like and rod-like shapes. The c -axis of α -SiC can be oriented parallel to the magnetic field direction, and the anisometric shape of SiC also makes it can be easily textured by hot working and

TGG [275–277]. The preferred orientation growth and texture formation in SiC ceramics are usually resulted from the phase transformation from β -SiC with the cubic crystal lattice to α -SiC with the hexagonal crystal lattice, and the phase transformation usually happens during HF of the sintered specimens at high temperature, which can greatly improve their mechanical properties [160,278]. Lee *et al.* [279] prepared textured SiC–YAG composites by HP and subsequent HF. The HPed specimen possesses equiaxed microstructure with random grain orientation, while the HFed specimen possesses texture microstructure with platelike grains, as shown in Fig. 38. The c -axis of SiC in the HFed specimen is oriented parallel to the pressure direction. The textured SiC–YAG specimen has more significantly anisotropic and higher flexural strength, fracture toughness, and Vickers hardness perpendicular (810 MPa, $5.9 \text{ MPa}\cdot\text{m}^{1/2}$, and 24.4 GPa) and parallel (1150 MPa, $4.4 \text{ MPa}\cdot\text{m}^{1/2}$, and 26.8 GPa) to the c -axis orientation compared with the untextured SiC–YAG specimen (600 MPa, $3.9 \text{ MPa}\cdot\text{m}^{1/2}$, 23.7 GPa, and 640 MPa, $3.8 \text{ MPa}\cdot\text{m}^{1/2}$, and 23.9 GPa).

Monoclinic zirconia can be oriented with the (100) direction parallel to the magnetic field and transforms to tetragonal zirconia after sintering. It was found that

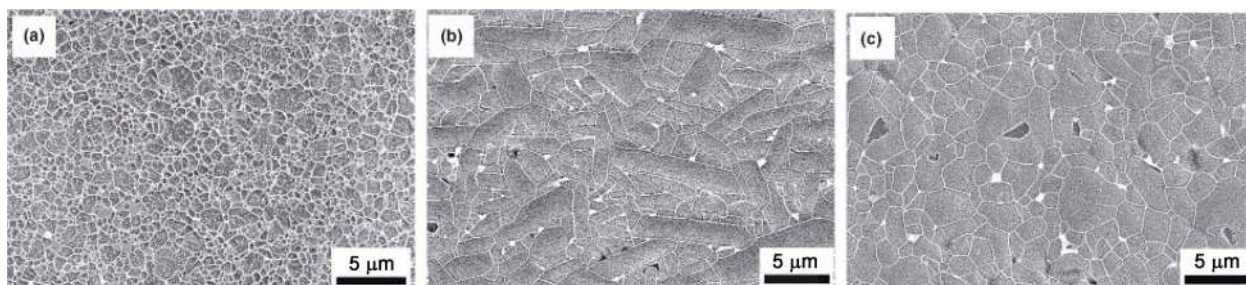


Fig. 38 Surface morphologies of the untextured HPed SiC–YAG specimen and the textured HFed SiC–YAG specimen: (a) HPed specimen surface parallel to the pressure direction; (b, c) HFed specimen surfaces parallel and perpendicular to the pressure direction, respectively. Reproduced with permission from Ref. [279], © Acta Materialia Inc. 2004.

the sintered tetragonal zirconia can inherit the alignment of the monoclinic zirconia particle, forming textured 3Y-TZP ceramics [280]. Vriami *et al.* [100] prepared textured 3Y-TZP ceramics using cubic 8 mol% yttria stabilized zirconia and unstabilized monoclinic zirconia as raw materials by magnetic alignment and pressureless sintering. The (001) direction of sintered tetragonal zirconia is parallel to the (100) direction of monoclinic zirconia. So the *c*-axis oriented 3Y-TZP ceramics are formed. But the textured specimen is almost entirely composed of equiaxed grains. Figure 39 shows the microstructure and Vickers indentations of untextured and textured zirconia ceramics. For the untextured specimen and the textured specimen surface perpendicular

to the magnetic field direction, crack propagations are isotropic along diagonal lines of the indentations, and the indentation toughness is 5.4 and 6.6 MPa·m^{1/2}, respectively. But for the textured specimen surface parallel to the magnetic field direction, there is hardly any crack propagation parallel to the *c*-axis orientation, and the crack propagation along the other direction (an indentation toughness of 11.8 MPa·m^{1/2}) is also much less obvious than that on the textured specimen surface perpendicular to the magnetic field. The anisotropy can be attributed to that martensitic transformation toughening mechanism is more predominant along the *c*-axis than perpendicular to the *c*-axis [281].

The texturing of B₄C ceramics can significantly

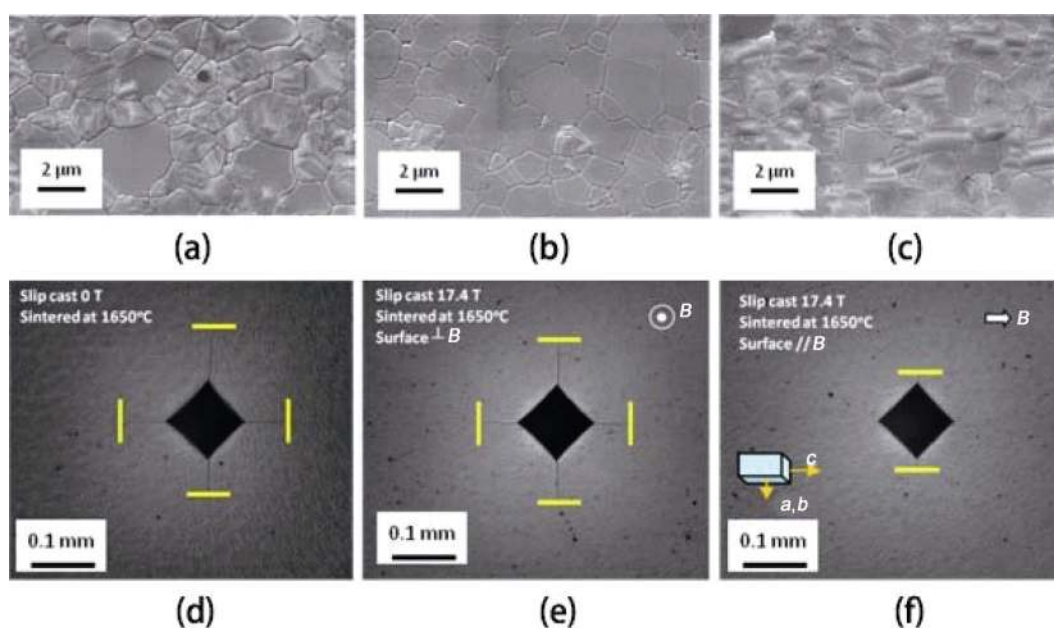


Fig. 39 Microstructure and 10 kg Vickers indentations of untextured and textured zirconia specimens: (a, d) untextured specimen; (b, c, e, f) textured specimen surfaces perpendicular (b, e) and parallel (c, f) to the magnetic field, respectively. Reproduced with permission from Ref. [100], © Elsevier Ltd. 2015.

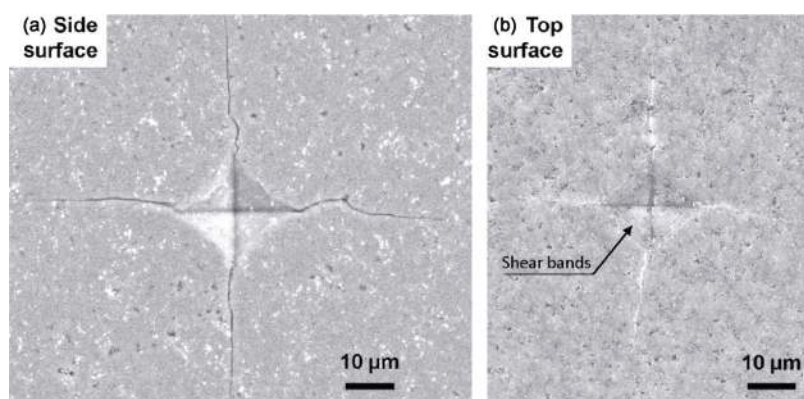


Fig. 40 Vickers indentations on textured B₄C specimen surfaces: (a) parallel to the *c*-axis orientation; (b) perpendicular to the *c*-axis orientation. Reproduced with permission from Ref. [283], © Acta Materialia Inc. 2010.

improve their hardness, even close to that of $B_{4.38}C$ monocrystal [274,282]. Grasso *et al.* [283] prepared textured B_4C ceramics by magnetic alignment and SPS. The Vickers indentations on the sintered specimen surfaces perpendicular and parallel to the c -axis orientation are shown in Fig. 40. The hardness measured on the surfaces perpendicular and parallel to the c -axis orientation is 36.7 and 33.8 GPa, respectively. The fracture toughness measured on the surfaces perpendicular and parallel to the c -axis orientation is 3.65 and 2.84 $MPa \cdot m^{1/2}$, respectively. The higher fracture toughness on the surface perpendicular to the c -axis orientation is attributed to the shear bands observed in fragments with thin edges, as shown in Fig. 40(b).

Structural ceramics can also be compounded to prepare textured composites possessing better properties. Poorteman *et al.* [284] prepared SiC platelets reinforced Si_3N_4 composites by HP. The SiC reinforced Si_3N_4 composites possess higher fracture toughness ($7.7 MPa \cdot m^{1/2}$) than that sintered without SiC platelets ($6.3 MPa \cdot m^{1/2}$) along the pressure direction. Wilk *et al.* [285] introduced h-BN to AlON ceramics by self-propagating high-temperature synthesis (SHS) and subsequent HP to improve the thermal conductivity of the composites. The texture degree of the composites reaches the highest with 30 wt% h-BN. The further increase of h-BN does not lead to higher texture degree due to the interlocking between plate-like h-BN grains. Thermal conductivities both parallel and perpendicular to the pressure direction reach the highest (13.9 and 42.0 $W/(m \cdot K)$, respectively) with 30 wt% h-BN.

5 Summary and outlook

The preparation methods and anisotropic properties of textured structural ceramics are reviewed. For ceramic materials, grains can be preferentially aligned along specific crystallographic directions under external physical field environments such as stress fields, electromagnetic fields, and temperature fields, forming texture microstructures. Several preparation methods of textured structural ceramics have been developed such as hot working, magnetic alignment, and TGG. Textured structural ceramics show obvious anisotropic properties and various damage mechanisms along different directions. Due to the grain orientation in textured structural ceramics, there are many strengthening

and toughening mechanisms such as crack deflection, crack bridging, and grain pull-out, resulting in higher strength and toughness along specific directions. The thermal conductivity along grain alignment direction is also higher than that perpendicular to the grain alignment direction due to the anisotropic phonon scattering. The good properties along specific directions of textured structural ceramics can greatly broaden their application fields.

Although much progress has been achieved, many issues are still not solved or need to be deeply investigated. For example, the dynamic mechanisms of preferred orientation growth of grains and its influence factors during preparation process of textured structural ceramics are still not clear, and the effects of phase transformation at high temperature on preferred orientation growth of grains, such as the transformation from α - Si_3N_4 to β - Si_3N_4 and from β -SiC to α -SiC, need to be studied. Besides, the combination of various texturing techniques, such as magnetic alignment and SPS, HP, and HF, have shown good potential in the preparation of ceramics with high texture degree. These and similar techniques need to be given more attention. Some textured composite ceramics and bioinspired ceramics with the hierarchical structure, which have shown excellent properties, need to be further developed.

There are many issues existing in this field, but there are also great potentials for optimizing the performances of textured structural ceramics. By further considering and investigating these issues, the microstructures and anisotropic properties of textured structural ceramics can be better controlled, and their application fields can be further expanded.

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