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Mechanical and thermal properties of 99% and 92% alumina at cryogenic temperatures

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Abstract

In the present paper, mechanical properties with respect to bending strength and fracture toughness of 99% and 92% Al_2O_3 at ambient and cryogenic temperatures (293 K, 195 K and 77 K) were measured with four-point bending method and SENB method respectively. It shows that the bending strength of both 99% and 92% Al_2O_3 seemed not to depend on temperature and with decreasing temperature there was a slight increase in the fracture toughness of 99% Al_2O_3 at 77 K but that of 92% Al_2O_3 remained almost unchanged with measurement uncertainties taken into account. It was also discovered that the reduction in the fracture toughness of glass phases in grain boundary had a negative effect on the improvement in that of α-Al_2O_3, thus the resultant toughness at 77 K appears not to increase as 99% Al_2O_3 does. On basis of qualitative analysis of phase compositions by XRD, the decrease in temperature down to 77 K did not result in any phase transformation of which the cryogenic mechanical properties are independent. The thermal conductivity of 99% and 92% Al_2O_3 was 4.1 W/(m K) and 1.7 W/(m K) at 20 K, respectively, much lower than that of stainless steel. Simultaneously alumina, whose thermal expansion is low between 123 K and 170 K, can be stable enough to temperature changes. Alumina ceramics, therefore, can be candidate materials for cryogenic application.

Keywords: Alumina; Cryogenic temperatures; Mechanical and thermal properties

1. Introduction

For cryogenic engineering, such as superconducting magnets, it is significant for the stable operation to reduce the heat penetration to the cryogenic area. According to the thermal design of large helical device (LHD), it has been clarified that the heat penetration through the supports, which are made of stainless steel, is dramatically larger than expected [1]. Thus it is necessary to select a material whose average thermal conductivity is small.

Since ceramic materials have good mechanical properties and favorable thermal properties (low thermal conductivity and thermal expansion) [2], they can be used as adequate materials in cryogenic engineering. However, only a small quantity of researches, mainly focused on Mg–PSZ [3,4], Ce–TZP [5], have been reported on the characteristics of ceramics at cryogenic temperature until now.

In the present paper, the mechanical and thermal properties of widely used alumina ceramics have been investigated at ambient and cryogenic temperatures. The purpose of this paper is to examine how the temperature and glass phases influence the properties and thereby the feasibility of utilizing alumina ceramics in cryogenic engineering.

2. Experimental procedures

Two kinds of sintered alumina ceramics, containing 99% and 92% α-Al_2O_3 (short for 99% Al_2O_3 and 92% Al_2O_3) respectively, were used for specimens. The bulk density measured by immersion in water using Archimede's principle were 3.89 g/cm^3 for 99% Al_2O_3 and 3.61 g/cm^3 for 92% Al_2O_3. The mechanical properties with respect to bending strength [6–8] and SENB-fracture toughness [9–11] at cryogenic temperatures were tested by low temperature test chamber and the universal testing machine (AG-IC, Shimadzu). At least six specimens for each material were tested and the average value was obtained for analysis. To ensure the accuracy of the operation in refrigerants, four-point bending tests were

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Table 1
Information about measurements of mechanical properties.

	Bending strength	SENB-fracture toughness
Method	Four-point bending	Four-point bending and SENB
Specimen size (mm)	3 × 4 × 36	6 × 4 × 36
Specimen requirements	Tensile face polished	Notched ($W = 0.2$ mm, $D = 3$ mm)[a]
Cross-head speed (mm/min)	0.5	0.05
Refrigerants	77 K	Liquid nitrogen
	195 K	Dry ice and ethanol

[a] W = width, D = depth.

performed. Information about measurements of mechanical properties was tabulated in Table 1.

Phase recognition and evaluation were made by XRD (D/max 2550HB, Rigaku). Microstructure of 92% Al_2O_3 was evaluated via SEM (JSM-6460LV, Shimadzu) images of polished and thermal-etched surfaces. The thermal conductivity was measured by PPMS (PPMS-9T, Quantum Design) in the range from 20 K to 360 K, and the thermal expansion was characterized by thermal expansion measuring instrument (DIL402C, Netzsch) ranging from 123 K to 283 K.

3. Results and discussion

Compared to ZrO_2-based toughening ceramics with the feature of martensitic tetragonal–monoclinic transformation toughening that phases and thus mechanical properties can be significantly affected by temperature [12], there exists no reports that the decrease in temperature can give rise to any phase transformations in alumina ceramics. In order to examine the difference in phase compositions at ambient and cryogenic temperatures, XRD was performed at ambient temperature for the surfaces of 99% and 92% Al_2O_3 fractured at 293 K, 195 K and 77 K respectively, as shown in Fig. 1. It is observed that 99% Al_2O_3 was almost completely α-Al_2O_3 while 92% Al_2O_3 consisted of α-Al_2O_3 and $MgAl_2O_4$ phase. In both materials, the decrease in temperature down to 77 K did not result in any transformation in phase compositions. SEM image of the thermal-etched surface of 92% Al_2O_3 and EDS analysis on grain boundary of the surface before being thermal-etched have been demonstrated in Fig. 2. It can be observed that the addition of sintering agent leads to the formation of glass phases in grain boundary composed of Mg, Si, Na, F (Fig. 2(b)), which have been removed by thermally etching so that the grains appear to be much more distinct. With the exception of Al_2O_3 phases (crystalline phases), the presence of glass phases, especially in 92% Al_2O_3, can also be a factor affecting the cryogenic mechanical properties.

The bending strength and fracture toughness data tested at 293 K, 195 K and 77 K are shown in Fig. 3. It can be seen from Fig. 3(a) that the bending strength of both 99% and 92% did not change so much within statistical discrepancy that the bending strength of 99% and 92% Al_2O_3 kept in the ranges of 379–394 MPa and 279–293 MPa, respectively. Thus it might be inferred that the presence of glass phases exerts almost no influence on bending strength at cryogenic temperatures. It can also be observed from Fig. 3(b) that with decreasing temperature there is a slight increase in the fracture toughness of 99% Al_2O_3 at 77 K but the fracture toughness of 92% Al_2O_3 at 77 K remains almost unchanged with measurement uncertainties taken into account. According to Chen [13], it can be attributed to the deterioration in toughness of glass phases at cryogenic temperatures. With the temperature decreasing to 77 K, the reduction in the fracture toughness of glass phases in grain boundary has a negative effect on the improvement in that of α-Al_2O_3, thus the resultant toughness at 77 K appears not to increase as 99% Al_2O_3 does.

From the above discussion, it can be concluded that the favorable mechanical properties of both types of alumina at ambient temperature, especially 99% Al_2O_3, can be preserved at cryogenic temperatures as well. Apart from the consideration

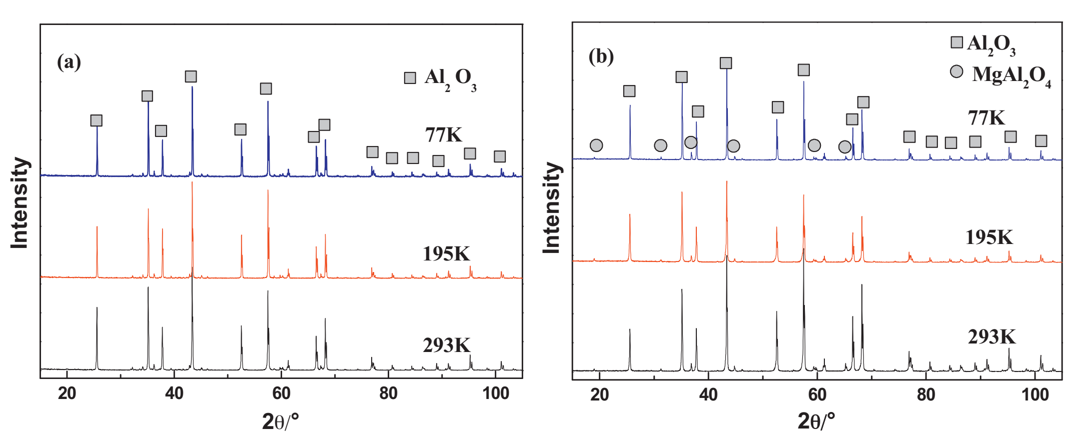

Fig. 1. XRD patterns of 99% and 92% Al_2O_3 fracture surfaces at 293K, 195K and 77K (a) 99% Al_2O_3; (b) 92% Al_2O_3.

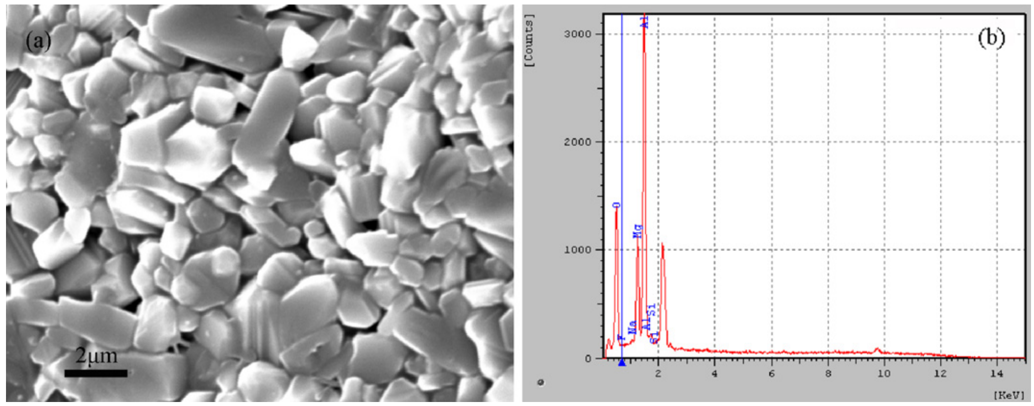

Fig. 2. SEM image and EDS analysis of 92% Al_2O_3. (a) SEM image of the thermal-etched surface; (b) EDS analysis on grain boundary of the surface before being thermal-etched.

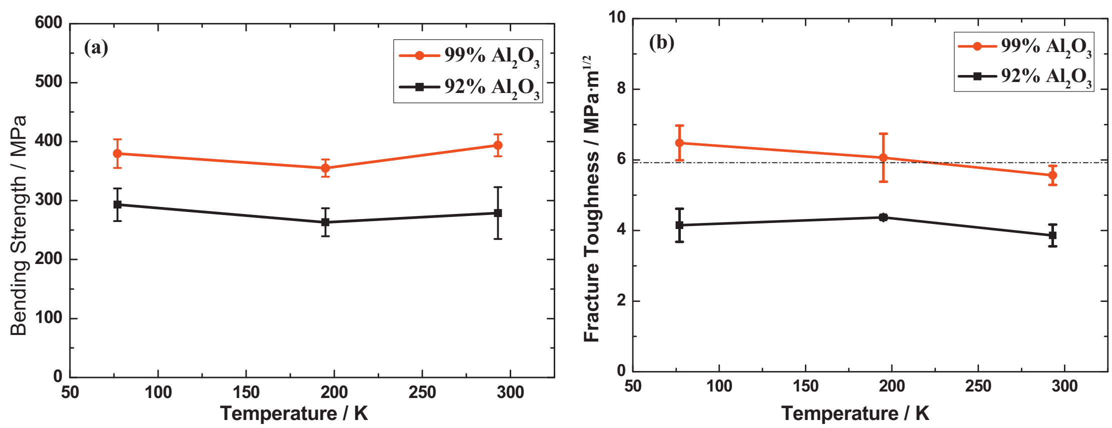

Fig. 3. Mechanical properties of 99% and 92% Al_2O_3 at 293K, 195K and 77K. (a) Bending strength; (b) fracture toughness.

of mechanical properties, another important factor to take into account is thermal properties with respect to thermal conductivity and thermal expansion. Fig. 4 shows the thermal conductivity and expansion of both types at various temperatures from 20 K to 360 K. It can be seen from Fig. 4(a) that the regularity of conductivity for 99% Al_2O_3 versus temperature is the same as 92% Al_2O_3 and both the values pass through a maximum in the vicinity of 110 K thereafter decrease dramatically with decreasing the temperature. The thermal conductivity of 99% and 92% Al_2O_3 drop to 4.1 W/(m K) and 1.7 W/(m K) at 20 K, respectively. At all test temperatures, the thermal conductivity of 99% Al_2O_3 proves to be larger than that of 92% Al_2O_3 owing to the presence of impurities and the resultant decrease of the mean free path of phonon of 92% Al_2O_3. It is worthy noting that the thermal conductivity of stainless steel in superconducting magnetic supports is 14.7 W/(m K) at cryogenic temperatures, much more larger than that of alumina ceramics in the present paper. Therefore, the heat penetration may be dramatically reduced in the case of utilizing alumina ceramics to superconducting magnetic supports.

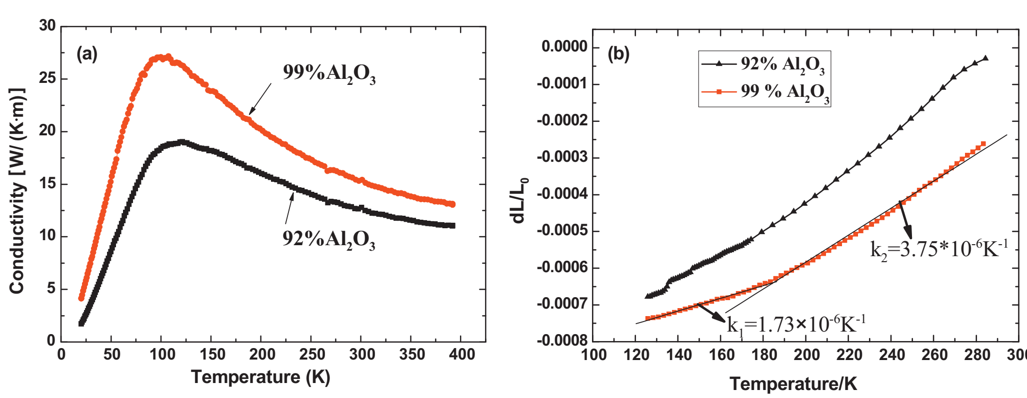

Fig. 4. The temperature dependence of thermal conductivity and expansion for 99%, 92% Al_2O_3. (a)Thermal conductivity; (b) thermal expansion.

High thermal expansion of a ceramic body can lead to substantial stresses and hence poor stability under conditions in which temperature varies to a great extent. It can be seen from Fig. 4(b) that the thermal expansion curve of 92% Al_2O_3 varied almost linearly with temperature while that of 99% Al_2O_3 exhibited an inflexion point at about 170 K. In general, an average value is sufficient for limited temperature ranges. On the basis of linear fit from Fig. 4(b), it can be calculated that the mean expansion coefficient of 92% Al_2O_3 from 123 K to 283 K is $4.07 \times 10^{-6}\ K^{-1}$ and those of 99% Al_2O_3 below and above 170 K are $1.73 \times 10^{-6}\ K^{-1}$ and $3.75 \times 10^{-6}\ K^{-1}$, respectively. All of such values prove to be low between 123 K and 170 K and thus alumina ceramics can be stable enough to temperature changes.

As a result, conclusion can be drawn that not only the mechanical properties but also the thermal properties of alumina ceramics can satisfy the requirements in cryogenic engineering and therefore be candidate materials for cryogenic application.

4. Conclusions

In conclusion, 92% Al_2O_3 has large quantities of glass phases which have almost no influence on bending strength of alumina ceramics at cryogenic temperatures. However, the negative effects of glass phases were responsible for the unchanged cryogenic fracture toughness of 92% Al_2O_3 which is reflected by the different change in fracture toughness of 99% and 92% Al_2O_3 at 77 K. It can be inferred from the measurement of thermal properties that the heat penetration may be dramatically reduced and the stability of superconducting magnetic supports can also be improved in the case of utilizing alumina ceramics to cryogenic engineering. The investigation indicated that alumina ceramics could be candidate materials for cryogenic application.

Acknowledgement

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