Study on the performance of alumina, zirconia and molybdenum composite insulation system for sapphire single crystal furnace

To cite this article: Jianpang Zhai et al 2018 IOP Conf. Ser.: Mater. Sci. Eng. 397 012057

View the article online for updates and enhancements.
Study on the performance of alumina, zirconia and molybdenum composite insulation system for sapphire single crystal furnace

Jianpang Zhai, Jianwei Jiang, Kaige Liu and Shuangchen Ruan

College of New Materials and New Energies, Shenzhen Technology University, Shenzhen 518118, P. R. China

Email: Jianpang Zhai.zhaijianpang@sztu.edu.cn

Abstract. In this paper, the properties of the combined temperature field of zirconia, alumina and metal have been studied by using the simulation software CGSim. For the first time, a thermal insulation system with the combination of molybdenum, zirconia and alumina screen has been proposed. By keeping the side of the screen thickness unchanged, the heat preservation effect of temperature field turns out to be worse when the molybdenum screen with the same thickness is replaced with alumina. To ensure internal molybdenum screen layers unchanged, the insulation effect of temperature field is significantly enhanced with increasing the thickness of alumina or zirconia, the power consumption is decreased. However, the decrease of power consumption slows down when the thickness of alumina or zirconia increase to a certain thickness.

1. Introduction

Due to its excellent physical and chemical properties, sapphire single crystal is widely used in satellite space technology, infrared devices, high-intensity laser window and other military fields [1–4]. Because of its lattice constant close to GaN and the growth process is mature, sapphire single crystal is widely used in SOS microelectronic circuits, LED lamp beads and other substrate industry [5, 6]. Owing to its high hardness, dielectric constant in line with the capacitive screen requirements, sapphire crystal can be used in the field of wearable equipment window [7-9]. Because of its special characteristics of the atomic level transition, titanium-doped sapphire single crystal was used in high-power laser gain media [10, 11]. With the rapid development of these industries, the growth of large-sized and high-quality sapphire single crystal has become particularly urgent [12-14]. As well known, the temperature field is the core part of the large-size sapphire single crystal furnace. The temperature field should not only have proper temperature gradient, solid liquid surface shape and melt liquid flow distribution, but also low power consumption. The selection of materials is crucial to the influence of power consumption. The traditional temperature field are made of high temperature resistant materials such as tungsten and molybdenum, its power consumption is relatively high. In recent years, the combined temperature field of ceramic and metal has been gradually adopted, which can effectively reduce the power consumption. However, it is not reported that the effect of combined temperature field of ceramic and metal on the temperature field. A reasonable and efficient design combined temperature field has always been a hard nut to crack for the development of the sapphire crystals industry.

CGSim software is an effective tool for the systematic study of the temperature field of sapphire single crystal furnace. In this paper, based on the insulation properties of molybdenum, zirconia and alumina, a thermal insulation system with the combination of molybdenum, zirconia and alumina
screen for 120 kg sapphire crystal furnace has been proposed. The effect of the matching between these three materials on the properties of temperature field has been studied. The final design can not only achieve the reasonable temperature gradient, good solid-liquid surface shape and melt flow distribution, but also achieve low power consumption.

2. Design Ideas of Temperature Field

The basis and process of CGSim software simulation in this article were described in literature [17]. In the design of the temperature field, we have adopted the following design steps as follows:

1) For the design of 120 kg thermal insulation system, molybdenum screen, zirconia, alumina fiber module were adopted as insulation material. The specific parameters of all kinds of materials were determined.

2) According to the crystal size, the specifications and dimensions of crucible and heating were determined, followed by zirconia and alumina fiber screen, finally the molybdenum screen size and the number of layers were determined.

3) The distance between different systems and the way of connection were constantly adjusted, so as to find a reasonable temperature field (temperature gradient, solid liquid surface, melt flow, temperature distribution and stress) on the basis of low power consumption.

Based on the above simulation, a reasonable temperature field was obtained, and then crystal growth experiment was carried out to verify the rationality of the temperature field.

**Figure 1.** The illustration structure of temperature field: 1 furnace shell; 2 seed bar; 3 heating; 4 upper thermal insulation system; 5 side thermal insulation system; 6 seed; 7 crucible; 8 crystal; 9 melt; 10 tungsten column; 11 bottom thermal insulation system.
3. Results and Discussion

3.1. Study on The side Thermal Insulation System

As shown in Figure 1, Cost savings and efficiency drove the design of side combination thermal insulation system (molybdenum screen and the alumina fiber module). The lower part of the side thermal insulation system will work for a long time in the high temperature area, which will be inevitably evaporated. The raw materials of alumina fiber module and sapphire crystal have the same ingredients, which can avoid contaminating the final crystal. In addition, the thermal insulation principle of alumina fiber and molybdenum screen is different: molybdenum screen insulation is the continuous reflection of thermal radiation, and the way of alumina thermal insulation is to block heat transfer from high temperature zone to low temperature zone. Considering that the thermal radiation is mainly in the high temperature region, and the heat conduction is mainly in the low temperature area, the molybdenum screen and the alumina fiber module are designed for the inner layer and outer layer, respectively.

To achieve the thickness of alumina and molybdenum screen, a simple theoretical simulation has been proposed. As shown in Figure 2, based on the thermal insulation of all molybdenum screen, the outer molybdenum screen was replaced by an equal thickness alumina fiber module. In order to study the introduction of alumina fiber module on the radial temperature distribution of the side thermal insulation system, according to the simulation results, the radial temperature distribution curve is plotted. As shown in Figure 3, the point A that is the internal side of the screen is defined as the
starting point, the point B that is the external side of the screen is defined as the end point. Then, a curve of the temperature distribution along the radial direction can be achieved.

The 0, 5, 8, 11, 15 and 20 layers of molybdenum screen were replaced by an equal thickness alumina fiber module, respectively. The radial temperature variation curve of side thermal insulation system can be shown in Figure 4. With the increase of the number of molybdenum screen replaced by alumina fiber module, the temperature of the radial point obviously increases, which indicates that the thermal insulation effect is worse. It can be explained as follows: In the inner region (high temperature area), the molybdenum screen continuously reflects the thermal radiation, which plays a good insulation effect, while in the outer region (low temperature area), the alumina fiber module obstruct the heat transfer from high temperature zone to low temperature zone. With the constant replacement of the molybdenum screen into the equal thickness alumina fiber module, the effect of the alumina fiber block on the heat is gradually enhanced, while the effect of the reflection of the thermal radiation on the molybdenum screen is gradually weakening. When the thickness of the alumina fiber module is not enough to compensate for the loss of heat caused by the reduction of the molybdenum screen, the thermal insulation effect of the whole temperature field will be worse. Therefore, it is particularly important to determine the number of molybdenum screen layers replaced by the same thickness of alumina.

From Figure 4, a very interesting phenomenon can be observed, at the same point in the temperature field, the temperature increases with the increase of the number of molybdenum screen layers replaced by the alumina fiber module. Considering the highest working temperature of alumina fiber module is 1600 degrees (1873K), and the increase of alumina thickness leads to the increase of temperature in the high temperature area of alumina fiber module (the outside of molybdenum screen). We choose the number of molybdenum screen layer as 10 layers (i.e., the distance from A point to 70mm and the 15 layer molybdenum screen is replaced by alumina). At this time, the temperature of alumina fiber module in high temperature area is 1770K, and the rising temperature interval of 100K is reserved for high temperature area of alumina.

![Figure 5](image)

**Figure 5.** Schematic diagram of alumina thickness: (a) 98.75mm; (b) 122.90mm; (c) 147.05mm; (d) 171.20mm; (E) 195.35mm

In order to study the effect of alumina thickness on the thermal insulation of whole temperature field, as shown in Figure 5, the thickness of the outer layer of alumina fiber module is gradually increased by keeping the 10 layers of the internal molybdenum screen unchanged. Considering the bottom insulation system as the full metal screen, the overall power consumption can be used as an index to evaluate the insulation capacity of the side screen. In other words, the thickness of alumina fiber module is determined by power consumption. Table 1 is the power consumption with different alumina thickness. It can be seen that the power consumption decrease with the increase of the thickness of alumina, the thermal insulation effect of the single crystal furnace is enhanced. However, the power consumption is on the increase when the thickness of alumina increase to 195.35mm, which is contrary to the experiment. This is because that the shock curve fitting is not convergent when the alumina thickness is 171.20mm. Based on the results of table 1, the side insulation system is set as follows: molybdenum screen with a thickness of 11 layer is placed inside of the side insulation system.
for high temperature; alumina fiber module with a thickness of 147.05mm is placed outside of the side insulation system for low temperature.

Table 1. Power consumption with the increase of alumina thickness

| alumina thickness (mm) | 98.75 | 122.90 | 147.05 | 171.20 | 195.35 |
|------------------------|-------|--------|--------|--------|--------|
| power consumption (kW) | 78.37 | 75.64  | 74.05  | 60.25  | 66.69  |

3.2. Study on the Bottom Thermal Insulation System

Because the working temperature of the bottom thermal insulation system is higher than that of the side insulation system, the molybdenum screen and the zirconia fiber module are set as the bottom thermal insulation system. According to the design process of the side insulation system, the outer molybdenum screen was replaced by an equal thickness zirconia fiber module. In order to study the introduction of zirconia fiber module on the axial temperature distribution of the bottom thermal insulation system, according to the simulation results, the axial temperature distribution curve is plotted. As shown in Figure 6, the point A that is the upper side of the screen is defined as the starting point of temperature curve, the point B that is the lower side of the screen is defined as the end point of temperature curve. Then, we can get a curve of the temperature distribution along the axial direction.

The 0, 5, 8, 11, 15 and 20 layer of molybdenum screen was replaced by an equal thickness zirconia fiber module, respectively. The axial temperature variation curve of bottom thermal insulation system can be shown in Figure 7. The temperature curve has little change at A point and B point, almost coinciding with the temperature curve. However, the difference appeared at the distance from B point 60mm. With the increase of the number of molybdenum screen layer replaced by zirconia, the temperature at the same point began to increase, and it began to coincide at the distance from B point 150mm. During crystal growth process, the crystallization temperature remains unchanged, thus A point keeps the same temperature. The cooling water inside the furnace shell takes away the remaining heat and the cooling water temperature is constant. The point B is in full contact with the furnace shell, thus B point also keeps the same temperature. The middle curve appeared temperature difference, at the same point in the temperature field, the temperature increases with the increase of the number of molybdenum screen layers replaced by the zirconia fiber module. However, the temperature dramatically decrease when the number of molybdenum screen layers with 26 replaced by the zirconia fiber module. When the number of replacement molybdenum screens is less than 26 layers, the thickness of zirconia is insufficient to make up for the loss of heat from the molybdenum screen. To increase the heat preservation, it is necessary to increase the thickness of the zirconia further.

![Figure 6](image1.png)

**Figure 6.** The position of temperature curve for the bottom thermal insulation system

![Figure 7](image2.png)

**Figure 7.** Temperature curves from point A to point B for bottom thermal insulation system
Table 2. Power consumption with the increase of zirconia thickness

| Zirconia thickness (mm) | 110.00 | 134.15 | 158.30 | 182.45 |
|------------------------|--------|--------|--------|--------|
| Power consumption (kW) | 76.38  | 73.61  | 72.60  | 71.98  |

Considering the highest working temperature of zirconia fiber module is 2000 degrees (2273K), and the increase of zirconia thickness leads to the increase of temperature in the high temperature area of zirconia fiber module (the outside of molybdenum screen). We choose the number of molybdenum screen layer as 10 layers (the distance from A point to 110mm). In order to study the effect of zirconia thickness on the thermal insulation of whole temperature field, the thickness of the bottom layer of zirconia fiber module is gradually increased by keeping the 10 layers of the upper molybdenum screen unchanged. Considering the side insulation system remaining unchanged, the overall power consumption can be used as an index to evaluate the insulation capacity of the bottom screen. In other words, the thickness of zirconia fiber module is determined by power consumption. Table 2 is the power consumption with different zirconia thickness. It can be seen that the power consumption decrease with the increase of the thickness of zirconia, the thermal insulation effect of the single crystal furnace is enhanced. However, the decreasing trend is slower when the thickness of the lower zirconia increases to 158.3mm, the power consumption has been reduced to 72.6kw. Thus the thickness of zircon is set as 158.3mm.

4. Conclusions

In this paper, a thermal insulation system with the combination of molybdenum, zirconia and alumina screen has been proposed. The molybdenum screen and the alumina fiber module are set as the side thermal insulation system. The heat preservation effect of temperature field turns out to be worse when the molybdenum screen with the same thickness is replaced with alumina. By keeping the molybdenum screen unchanged, thermal insulation increase with the increase of the thickness of alumina. The molybdenum screen and the zircon fiber module are set as the bottom thermal insulation system. The insulation effect of temperature field is significantly enhanced with increasing the thickness of zirconia, the power consumption is decreased. However, the decrease of power consumption slows down when the thickness of zirconia increase to a certain thickness.

Acknowledgment

This work was financed by the Research Project of Shenzhen Technology University 201707.

References

[1] Nassau K. Dr. A. V. L. Verneuil; The man and the method [J]. Journal of Crystal Growth, 1972, 13-14: 12-18.
[2] Harris D C 2003 A peek into the history of sapphire crystal growth Proc Spie50781-11
[3] Akasaki I 2007 Key inventions in the history of nitride-based blue LED and LD Journal of Crystal Growth, 300 (1)2-10
[4] Komaki H, Nakamura T, Katayama R, et al. Growth of In-rich InGaN films on sapphire via GaN layer by RF-MBE [J]. Journal of Crystal Growth, 2007, 301-302 (4): 473-477.
[5] Foxon C T, Campion R P, Grant V A, et al. Use of band-gap thermometry to investigate the growth of GaN on sapphire and GaAs [J]. Journal of Crystal Growth, 2007, 301 (4): 482-485.
[6] Yamada M, Tao C. Microscopic observation of strain induced in heteroepitaxial layers with reflection type of infrared polariscope [J]. Journal of Crystal Growth, 2000, 210 (1): 102-106.
[7] Dobrovinskaya E R, Lytvynov L A, Pishchik V. Sapphire: Material, Manufacturing, Applications [M]. Springer Publishing Company, Incorporated, 2009.
[8] Schmid F, Khattak C P, Rogers H H, et al. Current status of very large sapphire crystal growth for optical applications[J]. Proceedings of SPIE - The International Society for Optical Engineering, 1999: 70-76.
[9] Song C, Hang Y, Xia C, et al. Growth of composite sapphire/Ti:sapphire by the hydrothermal method[J]. Journal of Crystal Growth, 2005, 277 (1-4): 200-204.
[10] Schmid F, Viechnicki D J. Apparatus and method for unidirectionally solidifying high temperature material: U.S. Patent 3, 653, 432, [P]. 1972-4-4.
[11] Khattak C P, Schmid F. Growth of the world's largest sapphire crystals [J]. Journal of Crystal Growth, 2001, 225 (2-4): 572-579.
[12] Labelle H E. EFG, the invention and application to sapphire growth [J]. Journal of Crystal Growth, 1980, 50 (1): 8-17.
[13] Akselrod M S, Bruni F J. Modern trends in crystal growth and new applications of sapphire [J]. Journal of Crystal Growth, 2012, 360: 134-145.
[14] Budenkova O N, Vasiliev M G, Yuferev V S, et al. Simulation of global heat transfer in the Czochralski process for BGO sillenite crystals [J]. Journal of Crystal Growth, 2004, 266 (1–3): 103-108.