High-quality rectangular Y_2O_3-ZrO_2 single crystal optical waveguides for high-temperature fiber-optic sensors

Limin Tong* [a,b], Jingyi Lou** [a], and Eric Mazur [b]

[a] Department of Physics, State Key Laboratory of Silicon Materials, Zhejiang University, Hangzhou 310027, P.R.China

[b] Division of Engineering and Applied Sciences, Harvard University, Cambridge, MA 02138, USA

ABSTRACT

High quality Y_2O_3-ZrO_2 single crystal rectangular waveguides had been developed for high-temperature sensing applications. The waveguides were fabricated from bulky Y_2O_3 stabilized ZrO_2 single crystal by precise cut and fine polish. Three rectangular waveguides with cross-section larger than 1mm×1mm and length of 45mm～65mm were obtained. They showed much better optical properties than Y_2O_3-ZrO_2 single crystal fibers grown for fiber-optic sensing in previous work, optical losses of these waveguides were lower than 0.03dB/cm at wavelength of 900nm, and they were able to endure temperature as higher as 2300℃. All of them survived a 10g vibration test with average STF(strain to failure) of about 0.25%. Experimental results show that, these waveguides are promising for fiber-optic sensing for temperature above 2000℃.

Key words: Y_2O_3-ZrO_2 single crystal, polish, rectangular waveguide, high-temperature fiber-optic sensing

1.INTRODUCTION

Accurate temperature distribution is an important parameter for design and improvement of the turbine engine that widely used in aircrafts. Generally, the higher temperature the combustion achieves, the higher efficiency of the engine provides. The maximum temperature of the combustion can be near or even higher than 2000ºC and the high-temperature gas flow is oxidative, so we need fiber-optic sensor(it is much stable and accurate than other sensors in such environment) that is able to work at temperature higher than 2000°C. Although it is very successful in high temperature sensing, the currently used sapphire fiber-optic sensor can not be used at temperature higher than 1900 ° C due to the softening and melting of the sapphire fiber (melting point of sapphire crystal is 2045° C)[1], fiber materials with higher melting points must be used to substitute the sapphire. So we turn to Y_2O_3-ZrO_2 (Y_2O_3 stabilized ZrO_2), a cubic transparent crystal material with melting point of 2690 ° C.

*Contact: ltong@deas.harvard.edu; phone: 1 617 495 9616; fax: 1 617 496 4654; Harvard University, McKay Lab 327, 9 Oxford Street, Cambridge MA02138, USA; **lou-jy@263.net; phone: 86 571 87967187; fax: 86 571 87951358; Department of Physics, Zhejiang University, Hangzhou 310027, P.R.China.

As an optical waveguide for transmission radiation signal at high temperature, Y_2O_3-ZrO_2 single-crystal materials are promising for developing ultra-high-temperature fiber-optic sensors to work above 2000° C. In past work, Y_2O_3-ZrO_2 single-crystal in cylindrical fiber form has been used [2]. However, grown by conventional LHPG(Laser Heated Pedestal Growth) system, these fibers showed relatively low optical and mechanical qualities due to thermal stress caused by large temperature gradient during the growth[3,4]. Although, recently, Y_2O_3-ZrO_2 single-crystal fibers with higher transmittivity and less stress had been grown by means of an improved LHPG system[5], optical losses of these fibers(about 0.4dB/cm) were still too higher comparing with high-quality sapphire fibers(less than 0.04dB/cm). The high loss substantially prevents the Y_2O_3-ZrO_2 fiber-optic sensors from achieving excellent performances of sapphire fiber-optic sensors, the dominant high-temperature fiber-optic sensor for temperature below 1900 ° C.

Aiming to obtain high-quality Y_2O_3-ZrO_2 single-crystal waveguide with optical loss as low as sapphire fiber, in our work, rectangular Y_2O_3-ZrO_2 single crystal optical waveguides have been developed. By means of precise polish processes, the waveguides were fabricated from Y_2O_3-ZrO_2 single-crystal bars that were cut from an as-grown bulky crystal. Experimental results show that the average optical loss of these waveguides is less than 0.03dB/cm, which is similar to that of high-quality sapphire fibers and is much lower than that of Y_2O_3-ZrO_2 single-crystal fibers grown by LHPG method. With these waveguides, Y_2O_3-ZrO_2 high-temperature fiber-optic sensors with better performances could be developed.

2.FABRICATION OF Y_2O_3-ZRO_2 SINGLE CRYSTAL RECTANGULAR WAVEGUIDES

The Y_2O_3-ZrO_2 single crystal rectangular waveguides were fabricated by the following processes: First, cut an as-grown bulky cubic 21.2at.% Y_2O_3 stabilized ZrO_2 single crystal(grown by skull-melting method) into bars with cross section of about 1.5mm×1.5mm along the main axis, the lengths of the bars were 45~65mm, longer bars(such as 80mm) were available but they were very likely to break in grinding and polishing processes with our current facilities. Second, precisely grinded all surfaces (including end-faces) of the bars by an electric grinding machine with a 100-micron grit-fineness emery grit disc, until all obvious irregularities were removed. Then carefully polished the bars with diamond lapping films with grain-fineness of 30-µm, 15-µm, 5-µm, 3-µm, 1-µm and 0.5-µm in progression until optical-quality surfaces were obtained. Finally, removed polishing debris by an ultrasonic cleaner with methylbenzene, alcohol and acetone in succession. Three waveguides were fabricated with geometric parameters listed in Table.1, the photo of sample No.2 taken by a digital camera is shown in Fig.1. The waveguides were examined by a metalloscope, the major micro-defects observed were those with sizes smaller than 2µm, as shown in Fig.2, defects with sizes larger than 4µm is very scarce, and no defect with sizes larger than 10µm was found.

Table 1. Geometric parameters of Y_2O_3-ZrO_2 waveguides

Sample number	1	2	3
Length (mm)	45	52	65
Cross-section (mm×mm)	1.0×1.1	1.1×1.2	1.2×1.2

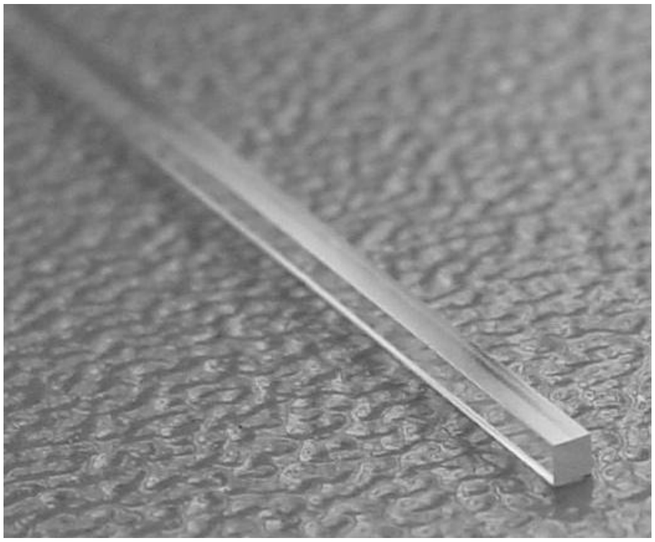

Figure 1: Photo of Y_2O_3-ZrO_2 rectangular waveguide No.2

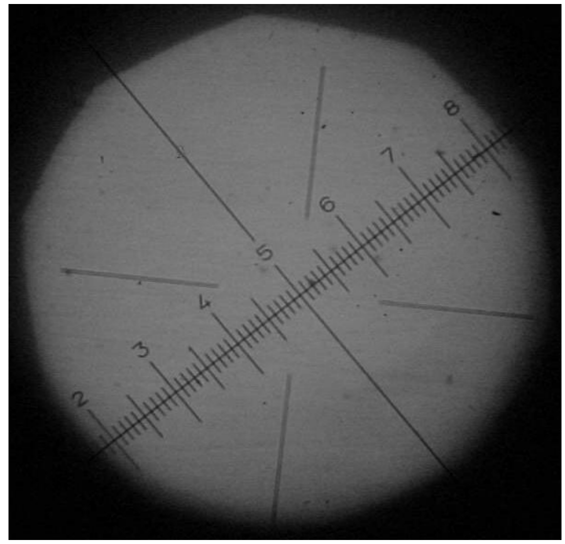

Figure 2: Micro-defects examination on the surface of waveguide No.2 under a metalloscope (500×)

3. PROPERTIES OF Y_2O_3-ZRO_2 SINGLE CRYSTAL RECTANGULAR WAVEGUIDES

3.1 Optical loss

The optical loss of the waveguide used for transmitting radiation signals is an important factor for temperature sensing. A high-loss waveguide will not only reduce the intensity of the useful signals, but also introduce noisy signals, degrading the performance(such as accuracy, resolution and working range) of the sensor. The purpose we fabricate the Y_2O_3-ZrO_2 waveguides here instead of Y_2O_3-ZrO_2 fibers is to reduce the optical loss of transmission waveguide and enhance the performance of the sensor.

In this work, the waveguides we obtained are relatively short, their transmission losses are very low and difficult to measure. So we improved our FM-1 single-crystal-fiber loss measurement system[6] as we did in other work [7]. As shown in Fig.3, a 150mm-long 450μm-thick sapphire fiber with intentionally grown defects at its center, was used as reference fiber. A photomultiplier with a 20mm-diameter integrating sphere was used to monitor the input power by scattering loss at the center of the reference fiber. A 50mm-long Y_2O_3-ZrO_2 single-crystal rectangular waveguides(fabricated by the same method as described above) with cross section of about 1.3mm×1.3mm fixed on another photomultiplier was used as reception waveguide. The testing waveguide was coupled to the reference fiber and reception waveguide by index matching liquid(index≈1.96). At the beginning, a shorter Y_2O_3-ZrO_2 rectangular waveguides with length of L_1 (~5mm) and almost the same cross-section as the testing waveguide was tested, recorded its output as P_1. Then the output of the testing waveguides with length of L_2 (45-65mm) was tested and recorded as P_2, assume the shorter waveguide had the similar loss behavior as the testing waveguide, the optical loss of the sample waveguide was obtained as

$$\alpha = \frac{L_2}{L_2 - L_1} \cdot 10 \cdot \lg \frac{P_1}{P_2}, \qquad (1)$$

by this design, the system could distinguish 0.1% difference (≈0.004dB) between P_1 and P_2 of the two waveguides. Ten measurements were made for each waveguide at the wavelength of 900nm, the average loss was shown in Fig.4. For comparison, optical losses of sapphire fibers and Y_2O_3-ZrO_2 single-crystal fibers grown by LHPG system in our previous work were also presented.

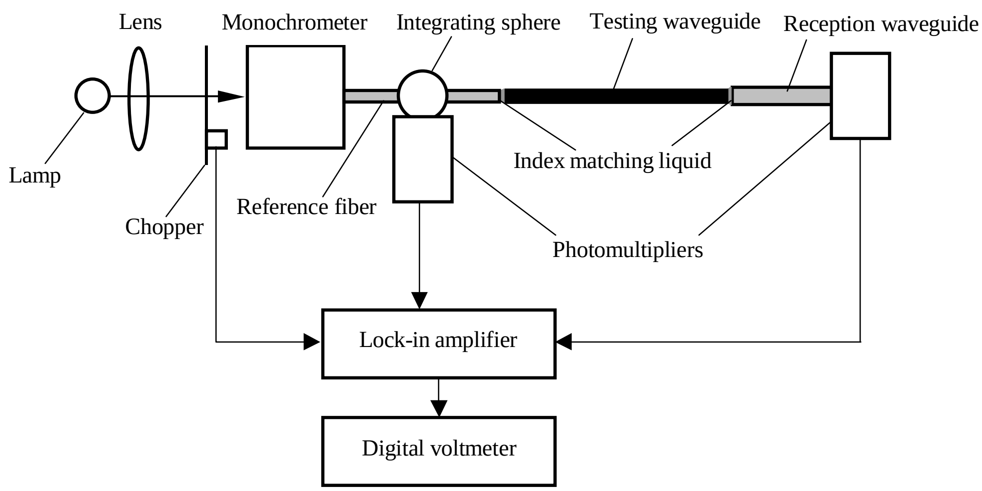

Figure 3: Improved loss measurement system for Y_2O_3-ZrO_2 rectangular waveguides

Results in Fig.4 show that, the average loss of these Y_2O_3-ZrO_2 waveguides is lower than 0.03dB/cm at 900nm, which is much lower than that of Y_2O_3-ZrO_2 fibers(about 0.4dB/cm) we have obtained in previous work[5] and is similar to that of sapphire fibers with similar cross-section (about 0.03 dB/cm). In addition, the Y_2O_3-ZrO_2 fibers are difficult to grown with diameter larger than 0.60mm due to stress-caused cracks, while Y_2O_3-ZrO_2 waveguides are easily to fabricate with large cross-sections. Generally, large cross-section area is beneficial to transmitting strong radiation signal, which may release the critical requirements of the photo-detection and signal processing systems especially when the signal is very weak.

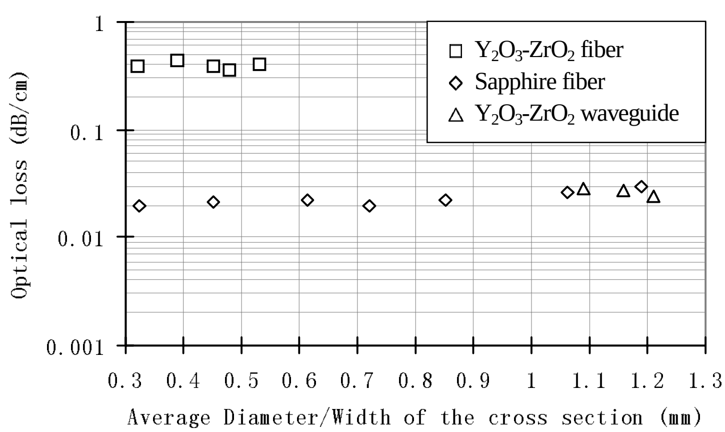

Figure 4 : Average losses of Y_2O_3-ZrO_2 waveguides, Y_2O_3-ZrO_2 fibers and sapphire fibers at wavelength of 900nm

Wavelength-dependent optical losses of these waveguides were also measured. Since the peak wavelength of black-body radiation at 2500℃(the assumed upper working temperature of the waveguide) is about 1.04μm according to Wien displacement law [8], the two detection wavelengths of a typical radiation-based two-band fiber-optic sensor for temperature lower than 2500℃ should be near but smaller than 1.04μm for high sensibility [1], therefore, in this work, we measured the waveguides within the wavelength range of 600～1000nm. Typical result of waveguide No.2 is shown in Fig.5, which shows that the loss of the waveguide within the measuring range(600～1000nm) is lower than 0.06dB/cm, which is favorable for radiation-based sensing. Similar results were obtained for other waveguides.

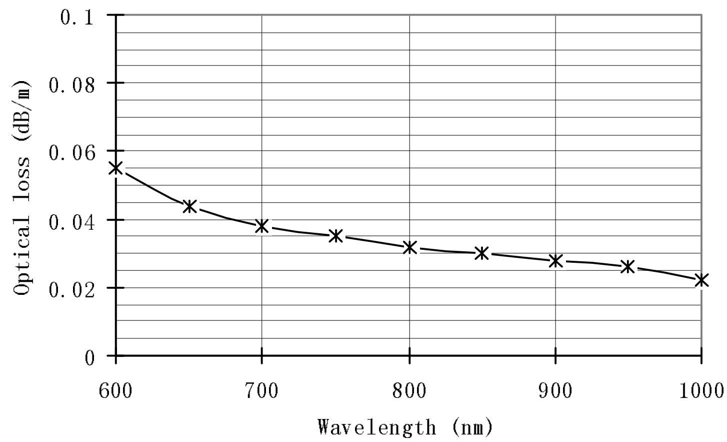

Figure 5: Wavelength-dependent optical loss of waveguide No.2 within 600～1000nm

3.2 High-temperature stability

As developed for high-temperature sensing, the high-temperature stability is very important for repeated uses. In this work, high-temperature stability of the sample No.2 was tested in a high-temperature furnace and an oxygen-alcohol-vapor flame in succession.

At first, the waveguide was placed on a zirconia ceramic substrate in the furnace, the temperature of the furnace was maintained at 1650℃ for 5 hours, we could not find any degradation of the waveguide after it was taken out from the furnace.

Then, the waveguide was tested in an oxygen-alcohol-vapor flame. Clamped one end of the waveguide, horizontally placed the other end in the flame, a sapphire fiber with similar size was juxtaposed for reference, a two-color pyrometer was used to monitor the end-temperature of the waveguide. The temperature of the flame was controlled by the rate of the oxygen flow. At first, no additional oxygen was provided, the temperature of the waveguide-end was about 800℃ in alcohol-vapor flame. Then, slowly increased the oxygen flux, when the temperature exceeded about 1900℃, the end part of reference sapphire fiber was softening and drooped. When the temperature exceeded 2000℃, the pendent end of the sapphire fiber was melted into a sphere, the highest temperature obtained in the test was about 2300℃. Details of the test are listed in Table.2.

Table.2. Details of the Y_2O_3-ZrO_2 waveguide No.2 tested in oxygen-alcohol-vapor flame

Y_2O_3-ZrO_2 waveguide	Dimension of sapphire fiber Diameter/Length (mm)	Beginning temperature (℃)	Soften temperature of sapphire fiber (℃)	Melting temperature of sapphire fiber (℃)	Highest temperature in the test (℃)	Total time lasted (minutes)
No.2	0.98/60	~800	1900～2000	2000～2100	~2300	～40

After the tests, no melting or obvious damage of the waveguides was found. The optical loss of the waveguide was measured again, the loss was about 0.030dB/cm, similar with the original loss of 0.029dB/cm before the test. Test results show that, the Y_2O_3-ZrO_2 waveguide is able to bear the temperature as high as 2300℃ without obvious degradation in optical properties, which can never be achieved by sapphire fibers.

3.3 Mechanical strength

Mechanical strength is important for waveguides in sensing applications, especially in those systems designed for measuring gas flow in turbine engines. In this work, mechanical strengths of these waveguides were tested by two methods. First, they were tested on a vibration platform, each with one end perpendicularly fixed and the other end freely impended. The test lasted about one hour with vibration frequency of 50Hz and an acceleration of about 10g(acceleration of gravity), all waveguides survived. Then, they were tested by 3-point bending to obtain the STF(strain to failure), which is a commonly used criterion for evaluating the mechanical strengths of fiber-like crystals[5,7,9,10]. The test was conducted in the air at room temperature(25℃) with relative humidity of 65%. Test results of STF are listed in Fig.6, for comparison, STF of Y_2O_3-ZrO_2 fibers of previous works[4,5] are also provided. Results show that, the average STF of the waveguides is about 0.25%, which is similar to that of improved fibers with much smaller cross-sections, Meanwhile, unlike fibers, whose STF decrease with the increasing diameter due to larger residual stress for larger diameter, these waveguides almost show a constant STF for different sizes, which indicate that increase widths of these waveguides does not lead to the reduction of their mechanical strengths. In fact, since they have not experienced the LHPG process with large growth rates as the fibers, and no additional stress is introduced during the grinding and polishing processes, the mechanical strength of these waveguides are mainly determined by the quality of the original crystal from which they were fabricated. In addition, STF of sample No.2 shows no obvious reduction comparing with others that had not been treated under high temperature, indicating no obvious degradation of mechanical strength of the waveguides after exposure to high temperature.

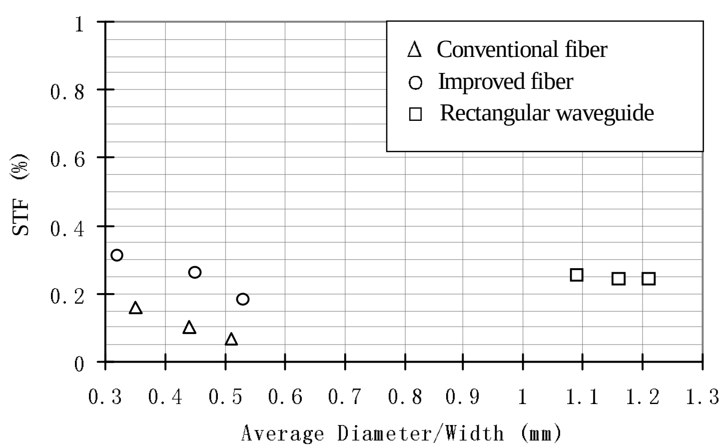

Figure 6: STF(strain to failure) of rectangular Y_2O_3-ZrO_2 waveguides and Y_2O_3-ZrO_2 fibers

4.CONCLUSIONS

Aiming to develop high-quality waveguides for temperature sensing, in this work, several Y_2O_3-ZrO_2 single crystal rectangular waveguides had been fabricated from a bulky cubic Y_2O_3 stabilized ZrO_2 single crystal. The waveguides have cross-sections large than 1mm×1mm and lengths of 45～65mm. At wavelength of 900nm, the average loss of these waveguides was as low as 0.03dB/cm, which is similar to that of high-quality sapphire fibers. High–temperature test show that the waveguide can survive temperature higher than 2300℃ without obvious degradation in optical and mechanical properties. High mechanical strength was also proved. In addition, this type of waveguide could be made with large cross-sections, which is beneficial to radiation-based sensing systems for obtain strong radiation signals. Based on their favorable optical and mechanical properties, Y_2O_3-ZrO_2 single crystal rectangular waveguides are promising for substituting Y_2O_3-ZrO_2 single-crystal fibers in ultra-high-temperature sensing applications.

Limited by the cut, grind and polish facilities, as well as the available size of the as-grown bulky Y_2O_3-ZrO_2 single crystals, the waveguides we obtained at present are relatively short for practical applications, and the lasting time for high-temperature test is not long enough. Keep this in mind, our further work on the Y_2O_3-ZrO_2 single crystal rectangular waveguides includes: improve the fabrication facilities, obtain longer waveguides(e.g. >100mm), and conduct a long-term high-temperature test of the waveguides. Meanwhile, fabricate waveguides with cross-sections other than rectangles, the edges of rectangular waveguides are too sharp, they are easily damaged, hexagonal or octagonal or even circular cross-sections will be better if they are fabricable.

ACKNOWLEDGEMENTS

This work is supported by National Natural Science Foundation of China (No.59906011) and 863 Youth Foundation of China(863-410-2000-5), we thanks Pao Yu-kong and Pao Zhao-long Scholarship for Chinese Students Studying Abroad, without its supporting, this work can not be completed.

REFERENCES

1. Limin Tong, “High-temperature single-crystal fibers and fiber-optic sensors for high-temperature”, PhD Thesis, Zhejiang University, Hangzhou, China, 1997.
2. Limin Tong, Yonghang shen, Linhua Ye and Zuchang Ding, “A zirconia single-crystal fibre-optic sensor for contact measurement of temperatures above 2000℃”, Measurement Science and Technology **10**, pp.607-611, 1999.
3. Limin Tong, Yanqi Wang and Zuchang Ding, “Growth and characteristics of Y-ZrO_2 SCF for high-temperature optic sensors”, Proc. SPIE **2292**, pp.429-438, 1994.
4. Limin Tong, “Study on growth and properties of Y_2O_3-ZrO_2 single-crystal fibers for fiber-optic sensors”, MS Thesis, Zhejiang University, Hangzhou, China, 1994.
5. Limin Tong, “Growth of high-quality Y_2O_3-ZrO_2 single-crystal optical fibers for ultra-high-temperature fiber-optic sensors”, Journal of Crystal Growth **217**, pp.281-286, 2000.
6. Mianyu Dong, Limin Tong and Zuchang Ding, “Loss measurement for SCF materials”, Proc. SPIE **2290**, pp.378-386, 1994.
7. Limin Tong, Yonghang Shen, Fangming Chen, and Linhua Ye, “Plastic bending of sapphire fibers for infrared sensing and power-delivery applications”, Applied Optics **39**, pp.494-501, 2000.
8. B.Yavorsky, and A.Detlaf, Handbook of Physics, Mir Publishers, Moscow, 1977.

9. G.N.Merberg and J.A.Harrington, "Optical and mechanical properties of single-crystal sapphire optical fibers", Applied Optics **32**, pp.3201-3209, 1993.
10. H.F.Wu, A.J.Perrotta and R.S.Feigelson, "Mechanical characterization of single-crystal α-Al_2O_3 fibres grown by the laser-heated pedestal technique", Journal of Materials Science Letters **10**, pp.1428-1429, 1991.