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Cover Page Footnote

The work was carried out within the framework of task 2.8 of the State Program of Scientific Research "Materials Science, New Materials and Technologies" for 2021–2025 in the Republic of Belarus. The authors are grateful to researcher T.D. Malikina for assistance in preparing samples and analyzing their phase composition, as well as to S.V. Grigoriev, leading engineer of the Polytechnic Scientific and Production Association of the Belarusian National Technical University, for recording X-ray diffraction patterns. Special thanks to the "Belmikroanaliz" State Center of OJSC "INTEGRAL" – INTEGRAL Holding Management Company for assistance in obtaining micrographs of the sample structure using a scanning electron microscope.

THE STRUCTURE AND PROPERTIES OF Er_2O_3 AND Yb_2O_3 BASED CERAMIC MATERIALS, SINTERED UNDER HIGH PRESSURE

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The aim of the work was to obtain high-density optically transparent ceramic samples by sintering cubic modification Er_2O_3 and Yb_2O_3 oxides micropowders at the pressure of 7 GPa and the heating current power of 0.6–1.0 kW, which was demonstrated. Non-monotonic dependence of the density on the heating current power has been established. The maximum density values of 9.2 g/cm^3 (Er_2O_3) and 9.8 g/cm^3 (Yb_2O_3) were achieved at the power of 0.8 and 0.9 kW, respectively. During sintering, the cubic phase partially transforms into the monoclinic phase, which is preserved in the obtained samples. The ceramics compaction process is accompanied by grain growth, more intense in erbium oxide samples. At sintering Yb_2O_3 micro-powder with particle size of 7.5–15 µm with the lowest heating current power value of 0.6 kW a microstructure similar to the nanocrystalline with the grain size of 90–200 nm, which contributes to higher transparency in the infrared wavelength range. Er_2O_3 samples have a discontinuous transmission spectrum, while Yb_2O_3 samples – a continuous one in the wavelength range of 400–850 nm. The maximum transmission values for ceramic samples are 29% at a wavelength of 750 nm, 52% at 849 nm for Er_2O_3 and 72% at 830 nm for Yb_2O_3.

Keywords: erbium oxide, ytterbium oxide, high pressure sintering, density, microstructure, phase composition, transparency

СТРУКТУРА И СВОЙСТВА КЕРАМИЧЕСКИХ МАТЕРИАЛОВ НА ОСНОВЕ ОКСИДОВ Er_2O_3 и Yb_2O_3, СПЕЧЕННЫХ ПОД ВЫСОКИМ ДАВЛЕНИЕМ

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Целью работы было получение высокоплотных оптически прозрачных керамических образцов спеканием микропорошков оксидов Er_2O_3 и Yb_2O_3 кубической модификации при давлении 7 ГПа и мощности тока нагрева 0,6–1,0 кВт, что и было продемонстрировано. Установлена немонотонная зависимость плотности от мощности тока нагрева. Максимальные значения плотности 9,2 г/см^3 (Er_2O_3) и 9,8 г/см^3 (Yb_2O_3) были достигнуты при мощности 0,8 и 0,9 кВт соответственно. При спекании кубическая фаза частично переходит в моноклинную, которая сохраняется в полученных образцах. Процесс уплотнения керамики сопровождается ростом зерна, более интенсивным в образцах оксида эрбия. При спекании микропорошка Yb_2O_3 с размером частиц 7,5–15 мкм при самом низком значении мощности тока нагрева 0,6 кВт формируется микроструктура, близкая к нанокристаллической, с размером зерна 90–200 нм, что способствует более высокой прозрачности в ИК диапазоне длин волн. Для образцов Er_2O_3 характерен прерывистый спектр пропускания, а для Yb_2O_3 непрерывный в диапазоне длин волн 400–850 нм. Максимальные значения пропускания керамических образцов составляют 29% при длине волны 750 нм, 52% при 849 нм для Er_2O_3 и 72% при 830 нм для Yb_2O_3.

Ключевые слова: оксид эрбия, оксид иттербия, спекание под высоким давлением, плотность, микроструктура, фазовый состав, прозрачность

Er_2O_3 VA Yb_2O_3 YUQORI BOSIMDAGI SINTERLANGAN OKSIDLAR ASOSIDAGI KERAMIK MATERIALLARNING TUZILISHI VA XUSUSIYATLARI

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Ushbu tadqiqotning maqsadi 7 GPa bosim va 0,6-1,0 kVt isitish oqimida kubik Er_2O_3 va Yb_2O_3 oksidlarining mikrochanglarini sinterlash orqali yuqori zichlikdagi, optik shaffof keramika namunalarini ishlab chiqarish edi. Isitish oqimiga zichlikning monoton bo'lmagan bog'liqligi aniqlandi. 9,2 g / sm^3 (Er_2O_3) va 9,8 g / sm^3 (Yb_2O_3) maksimal zichlik qiymatlariga mos ravishda 0,8 va 0,9 kVt isitish oqimida erishildi. Sinterlash jarayonida kub faza qisman monoklinik fazaga aylanadi, bu esa olingan namunalarda saqlanadi. Seramika zichlash jarayoni donning o'sishi bilan birga keladi, bu erbium oksidi namunalarida kuchliroqdir. Yb_2O_3 mikrochangini zarracha hajmi 7,5-15 mkm bo'lgan 0,6 kVt eng past isitish oqimida sinterlashda nanokristalga yaqin mikro tuzilma hosil bo'ladi, don o'lchami 90-200 nm bo'lib, bu IQ to'lqin uzunligi diapazonida shaffoflikni oshirishga yordam beradi. Er_2O_3 namunalari uzluksiz uzatish spektri bilan tavsiflanadi, Yb_2O_3 esa 400-850 nm to'lqin uzunligi oralig'ida uzluksiz spektrga ega. Keramika namunalarining maksimal o'tkazuvchanlik qiymatlari 750 nm to'lqin uzunligida 29%, Er_2O_3 uchun 849 nm da 52% va Yb_2O_3 uchun 830 nm da 72% ni tashkil qiladi.

Kalit so'zlar: erbiy oksidi, iterbiy oksidi, yuqori bosimli sinterlash, zichlik, mikro tuzilma, faza tarkibi, shaffoflik

Introduction

In recent years there has been a significant increase in the interest in producing optical ceramics that is based on oxides of various elements instead of the more expensive single crystals. Such ceramics have a number of advantages: the cost effectiveness of the production method, better mechanical properties, reduction of production time, possibility to control the form and organization of the large-scale production. Essential requirements for obtaining optical ceramics are high density, absence of the second phase at the boundaries of the grains or pores, small size of grains as compared to the visible light wavelength, and isotropic crystal lattice. The main ways of manufacturing such ceramics are hot pressing, hot isostatic pressing, vacuum sintering, spark plasma sintering, microwave sintering [1–4].

High pressure sintering method has a number of advantages over the other sintering methods. It does not require introduction of activating additives, ensures a high degree of initial powder compaction, requires less sintering time and provides for the formation of the nanocrystalline structure of the material being sintered [5, 6].

At present the promising compounds for production of optical ceramic materials for various uses are refractory REE sesquioxides due to their polymorphism and possibility of up to five different types of crystal lattices [7–17]. The most characteristic types are A-hexagonal, B-monoclinic and C-cubic lattice [18, 19]. Under the normal conditions they usually have isotropic cubic lattice. When heated, cubic modification turns into monoclinic modification. There is evidence of the phase transitions in such materials directly under high pressure [20–25] and after the thermal treatment at ultra-high pressures [26–28]. Nevertheless, there is practically no information on the structure formation and properties of ceramics based on these oxides sintered under high pressure.

The objective of study is to examine the possibility of producing transparent ceramics based on erbium and ytterbium oxides by high pressure sintering and to study their microstructure and properties.

Research methods

The starting materials were erbium and ytterbium oxide powders with a purity of 99.5 % and 99.92 % respectively. The powders were pre-annealed in a SNOL 6.7/1300 muffle furnace at a temperature of 1000 °C for 2 h. Samples with a diameter of 6.3 mm and a height of 4.3 mm were pressed in a steel mold at a pressure of 500 MPa. Sintering of the samples was carried out at a pressure of 7 GPa and a heating current of 0.6–1.0 kW in a high-pressure apparatus for 30 s [29]. For this aim, a DO137A pressing unit with a force of 5 MN, equipped with a KS-5 automated sintering mode control system, was used [30].

The density was determined by hydrostatic weighing in CCl_4.

The crystal structure was investigated by X-ray diffraction (XRD) measurements using DRON-7 diffractometer.

Electron microscopic studies were carried out using a scanning electron microscope S-4800 «Hitachi» (Japan).

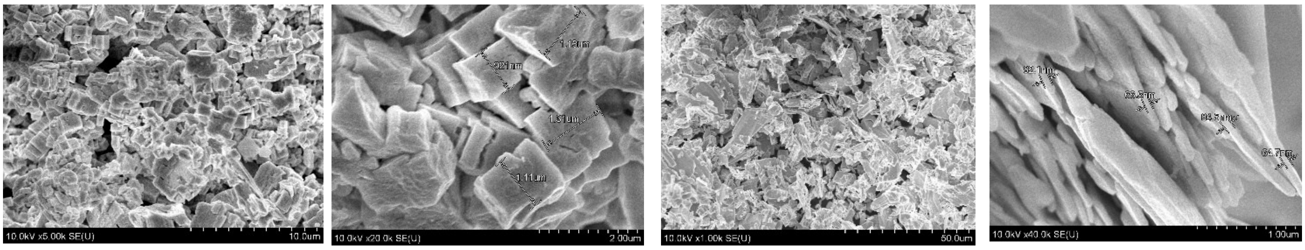
The optical transmission spectra were measured using an MDR-23U monochromator. A R91100 photomultiplier (Hamamatsu, Japan) with a spectral sensitivity range of 200–850 nm was used as a radiation detector. A 170-W tungsten incandescent lamp (KMC-17, Russia) served as a light source. The signal from the photodetector was fed to a preamplifier and then to a Unipan 232 B narrowband amplifier. Electrical signals were recorded at a frequency of ~20 Hz, followed by conversion of the main and reference signals into DC voltage using the phase detection method.

Results and Discussion

Fig. 1 shows the microphotographs of Er_2O_3 and Yb_2O_3 initial powders with different magnifications. Er_2O_3 powder particles are a collection of acute-angled blocks predominantly in the form of parallelepipeds ranging from 920 nm to 1300 nm in size. Yb_2O_3 powder particles have spiky-lamellar shape with the size of plate of 7.5–15 μm and thickness of ~ 65–85 nm.

Figure 2 shows the dependence of the density of sintered erbium and ytterbium oxides samples on the heating current power. Electron micrographs of sintered samples splits are shown in Figure 3 and Figure 4.

The change in density at the rise of the sintering temperature of Er_2O_3 and Yb_2O_3 samples is non-monotonic (Fig. 2). For Er_2O_3, the maximum density value of 9.2 g/cm^3 is reached at W = 0.8 kW, and that for Yb_2O_3 of 9.8 g/cm^3 – at W = 0.9



a b c d

Figure 1. Microphotographs of Er_2O_3 (a, b) and Yb_2O_3 (c, d) initial powders with different magnifications ***a*** **– x5.000;** ***b*** **– x20.000; c– x1,000;** ***d*****– x40.000.**

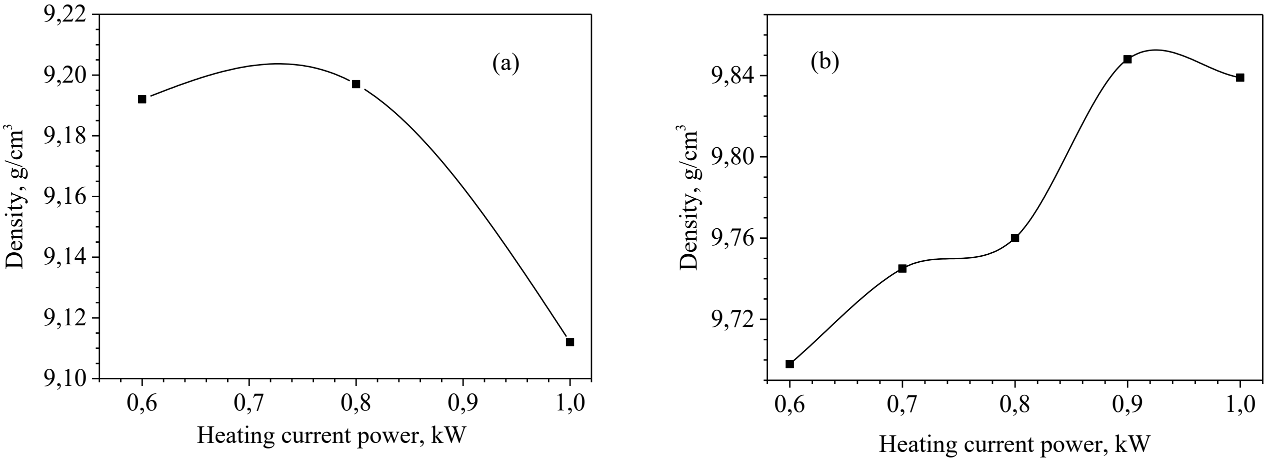
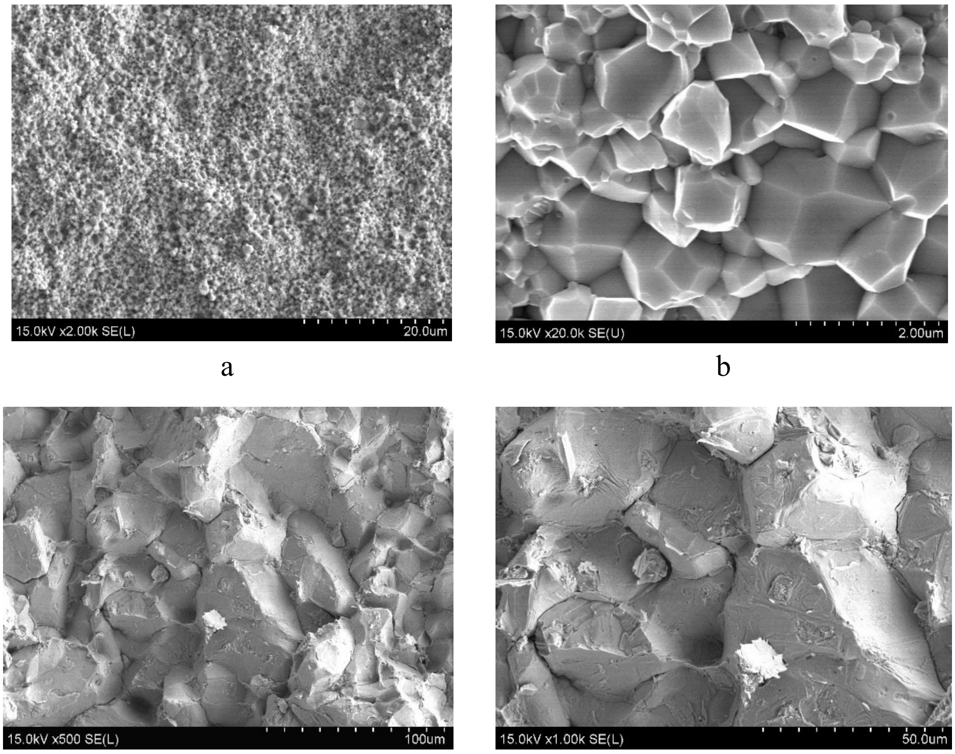


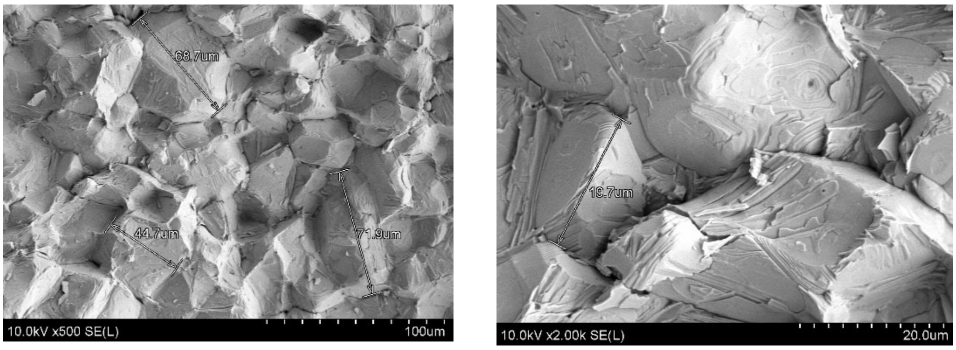
Figure 2. Dependence of the density of sintered Er_2O_3 (a) и Yb_2O_3 (b) samples on the heating current power.

kW due to the powder compaction, the initial cubic phase conversion into the monoclinic phase, and the recrystallization processes. The heating current power W increase during erbium oxide samples sintering leads to the intensive grain growth. At the heating current power of 0.6 kW, the grain size is 310–1740 nm (Fig. 3, a, b), and at 0.8 kW and 1.0 kW its value reaches 10–54 μm



a b

c d



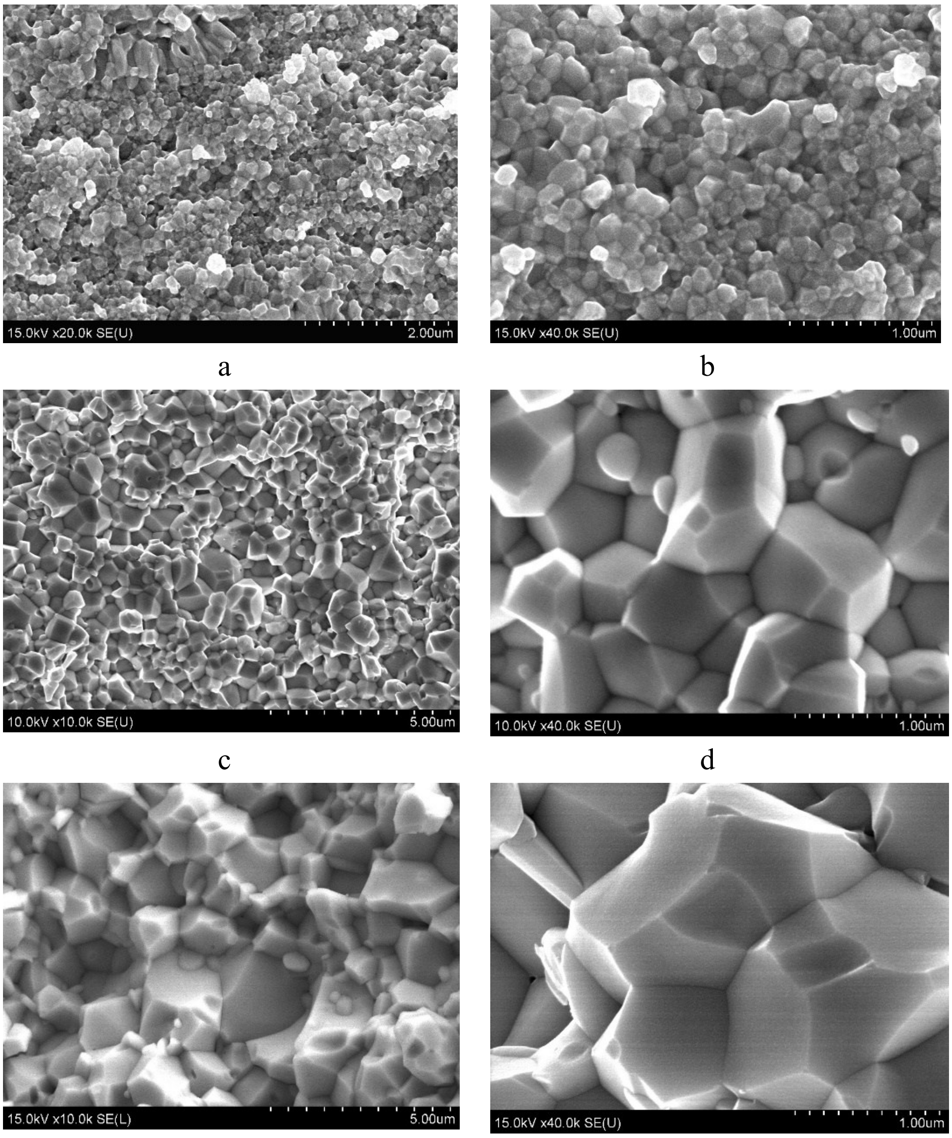
e f

Figure 3. Microfractograms of Er_2O_3 samples sintered at a heating current power of 0.6 kW (a, b); 0.8 kW (c, d); 1.0 kW (e, f) at different magnifications: a, f – x2,000; b – x20,000; c, e – x500; d – x1,000.

(Fig. 3, c, d) and 20–72 μm (Fig. 3, e, f) respectively.

The analysis of microphotographs of fractures (Fig. 3 and Fig. 4) shows the intercrystallite type of destruction of erbium and ytterbium oxides samples that were sintered at low heating current power (Fig. 3, c–f), and transcrystallite at high heating current power values (Fig. 4, g, h).

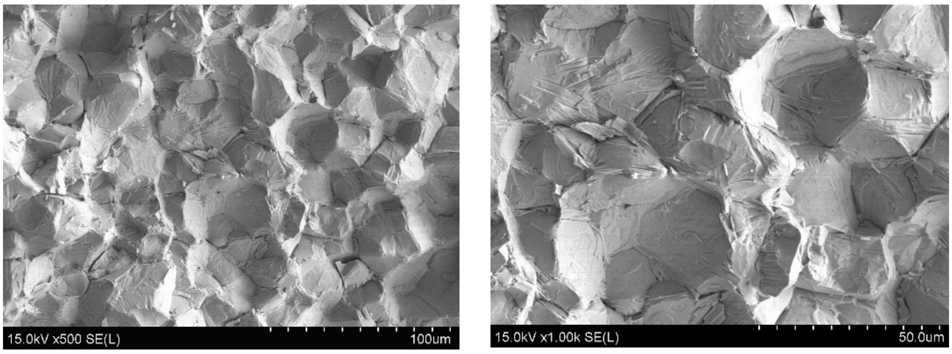
Figures 5a and 5b shows diffractograms of initial powders and sintered Er_2O_3 and Yb_2O_3 sam-



a b

c d

e f



g h

Figure 4. Microfractograms of Yb_2O_3 samples sintered at a heating current power of 0.6 kW (a, b); 0.7 kW (c, d); 0.8 kW (e, f); 1.0 kW (g, h) at magnification: a – x20,000; b, d, f – x40,000; c, e, – x10,000; g – x500; h – x1,000.

ples. These data prove that initial erbium and ytterbium oxide powders are single-phase and contain only cubic modification. During high-pressure sintering, a part of cubic modification is converted into monoclinic one, which is preserved in obtained samples. In addition, sintered yttrium oxide samples contain a small amount of Yb_3O_4 and YbO oxides (Fig. 5, b).

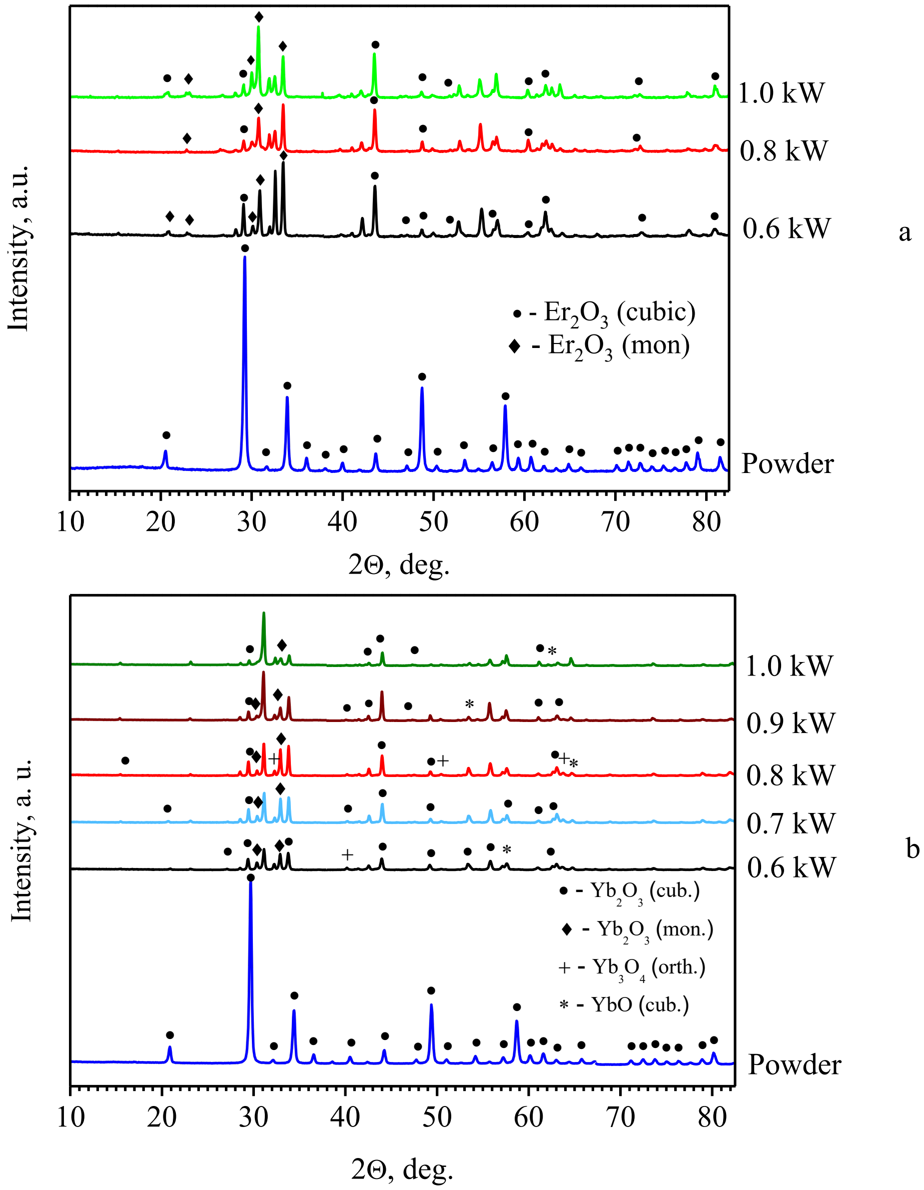


Figure 5. XRD patterns of initial powders and sintered Er_2O_3 (a) and Yb_2O_3 (b).

The transmission spectra in the wavelength range of 400–850 nm are shown in the Fig. 6. You can see that Er_2O_3 has a discontinuous transmission spectrum and Yb_2O_3 – a continuous transmission spectrum. The highest transmittance values are in the red and infrared regions of the spectrum for the samples with the lowest heating current power of 0.6 kW. This is due to low grain size (Fig. 3, a, b and Fig. 4, a, b) and lower content of monoclinic phase oxide (Fig. 5). For Er_2O_3 samples, transmission is 29 % and 52 % at the wavelength of 750 nm and 849 nm (Fig. 6, a). Yb_2O_3 samples have a higher transparency in the infrared range – 72 % at 830 nm (Fig. 6, b).

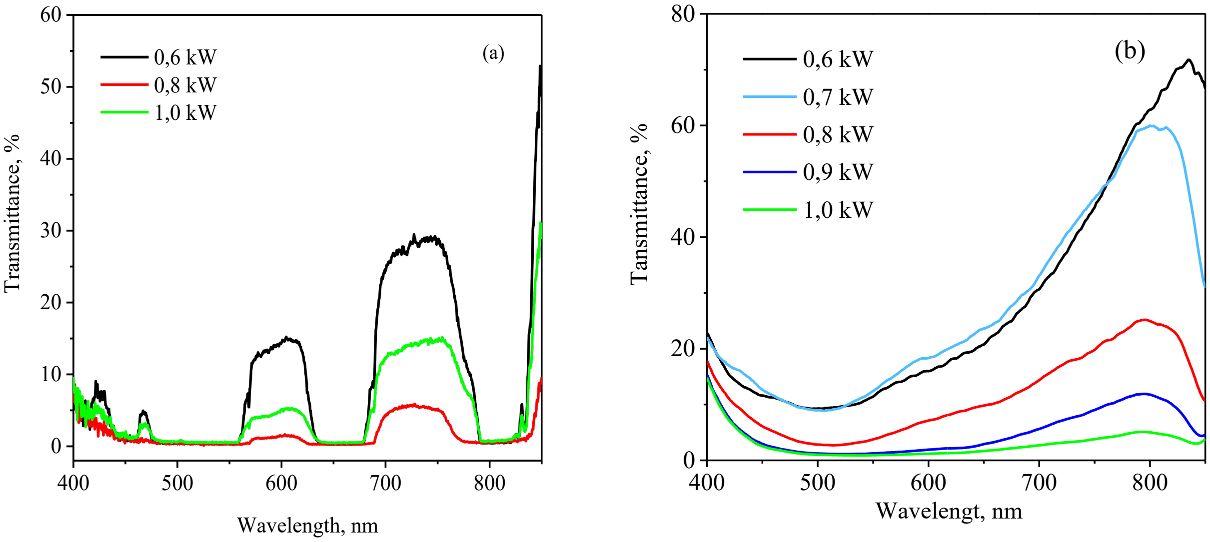


Figure 6. The transmission spectra of sintered Er_2O_3 (a) and Yb_2O_3 (b).

Conclusion

The possibility of obtaining high-density optically transparent ceramic samples by sintering cubic modification Er_2O_3 and Yb_2O_3 oxides micro-powders at the pressure of 7 GPa and the heating current power of 1.0 kW in a high-pressure apparatus was shown.

Non-monotonic dependence of density on the heating current power has been established. The maximum density values of 9.2 g/cm^3 (Er_2O_3) and 9.8 g/cm^3 (Yb_2O_3) were achieved at 0.8 and 0.9 kW, respectively. It is shown that at sintering partial polymorphic transformation occurs by the cubic modification of initial powder oxide into monoclinic. The sintering process is accompanied by grain growth, which is more intense in the erbium oxide samples.

It has been found that the type of destruction of sintered oxide samples with the sintering temperature increase changes from intercrystallite to transcrystallite. It has been established that at sintering Yb_2O_3 micropowder with particle size of 7.5–15 μm at low temperature, dense microstructure similar to nanocrystalline, with grain size of 90–200 nm, which facilitates higher transparency in the infrared wavelength range. Er_2O_3 has a discontinuous transmission spectrum, and Yb_2O_3 has a continuous transmission spectrum. The maximum transmission values for ceramic samples are 29% at a wavelength of 750 nm, 52% at 849 nm for Er_2O_3 and 72% at 830 nm for Yb_2O_3.

Acknowledgments

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