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Synthesis of high-entropy $(Y_{0.2}Yb_{0.2}Lu_{0.2}Eu_{0.2}Er_{0.2})_3Al_5O_{12}$ ceramics using a fast electron beam

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Abstract. High-entropy ceramics intended for thermal barrier coatings are developed to improve their performance properties, particularly by increasing their operating temperature. However, conventional synthesis of high-entropy ceramics is time-consuming. This study explores a nonconventional approach to reducing synthesis time by processing ceramic materials with a high-power beam of high-energy electrons (fast electrons). A powder mixture of the initial reactants Y_2O_3 , Yb_2O_3 , Lu_2O_3 , Eu_2O_3 , Er_2O_3 , and Al_2O_3 was heated in air using 1.4 MeV electrons at different electron-beam currents. The cuvette containing the powder mixture was moved beneath the beam at 1 cm/s, while the beam was scanned across the width of the internal volume of the cuvette. The total irradiation time was 10 s. At beam currents of 4 mA or higher, melt droplets formed within the irradiated powder mass, and their proportion relative to the unmelted powder increased with increasing current. Crystallization occurred in the melt droplets during cooling. The resulted droplet-shaped ceramic product was highly porous because of the intense release of adsorbed gases from the melt. SEM images and EDS elemental maps revealed a uniform distribution of the constituent elements throughout the droplet-shaped ceramic product. XRD analysis identified the synthesized material as high-entropy $(Y_{0.2}Yb_{0.2}Lu_{0.2}Eu_{0.2}Er_{0.2})_3Al_5O_{12}$ ceramic. The powder that did not contribute to the formation of the droplet-shaped product was an intermediate product containing $Er_3Al_5O_{12}$ and $Y_3Al_5O_{12}$ garnets, together with Er_2O_3 , Yb_2O_3 , Y_2O_3 , Eu_2O_3 , Lu_2O_3 , Al_2O_3 oxides.

Keywords: high-entropy ceramics, synthesis, high-power electron beam

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Синтез высокоэнтропийной керамики $(Y_{0.2}Yb_{0.2}Lu_{0.2}Eu_{0.2}Er_{0.2})_3Al_5O_{12}$ в пучке быстрых электронов

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Аннотация. При получении высокоэнтропийной керамики для использования в термобарьерных покрытиях ставится цель улучшения их эксплуатационных свойств, в частности увеличение температуры эксплуатации. Синтез высокоэнтропийной керамики является длительным процессом. В настоящей работе задача уменьшения временных затрат на синтез решается за счет применения нетрадиционного метода обработки керамических материалов мощным пучком высокоэнергетических электронов (быстрых электронов). Нагрев порошковой смеси исходных реагентов (Y_2O_3 , Yb_2O_3 , Lu_2O_3 , Eu_2O_3 , Er_2O_3 , Al_2O_3) быстрыми электронами с энергией 1,4 МэВ проводили на воздухе при различных значениях тока электронного пучка.

Скорость перемещения кюветы с порошковой смесью под пучком составляла 1 см/с. Сам пучок сканировали по ширине внутреннего объема кюветы. Общее время нахождения кюветы под пучком – 10 с. Установлено, что при токе ≥ 4 мА в облучаемой порошковой массе образуются капли расплава. Их доля по отношению к нерасплавленному порошку возрастает по мере повышения величины тока. В каплях расплава во время охлаждения происходят процессы кристаллизации. Синтезированный каплевидный керамический продукт имеет высокую пористость вследствие активного газовыделения адсорбированных газов в расплаве. СЭМ-изображения и ЭДС-карты показали равномерное распределение исходных элементов по объему каплевидного керамического продукта. Согласно РФА, материал синтезированной керамики представляет из себя высокоэнтропийную керамику ($Y_{0.2}Yb_{0.2}Lu_{0.2}Eu_{0.2}Er_{0.2}$)₃Al₅O₁₂. Порошок, не участвовавший в образовании каплевидного продукта, является промежуточным продуктом, содержащим гранаты $Er_3Al_5O_{12}$, $Y_3Al_5O_{12}$ и оксиды Er_2O_3 , Yb_2O_3 , Y_2O_3 , Eu_2O_3 , Lu_2O_3 , Al_2O_3 .

Ключевые слова: высокоэнтропийная керамика, синтез, мощный электронный пучок

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Introduction

Ceramics, owing to their unique properties, are frequently employed as alternatives to metals and alloys [1]. Where requisite mechanical strength cannot be achieved without metals, ceramics serve as protective coatings, providing enhanced heat resistance for the component as a whole [2; 3]. Such thermal barrier coatings (TBCs) are particularly vital in the aviation industry [4]. A TBC system typically comprises multiple layers [5], where the ceramic topcoat ensures thermal protection for components such as gas turbine blades [6; 7]. Traditionally, zirconium dioxide serves this purpose [8; 9]. Since this ceramic exhibit intrinsic polymorphism [10; 11], yttrium oxide is added to stabilize its crystal structure [12]. However, these measures remain inadequate for operation at elevated temperatures.

High-entropy ceramics (HECs) have recently been considered by researchers as an alternative to partially or fully stabilized zirconia [13–16]. HECs are solid solutions of inorganic compounds characterized by one or more Wyckoff sites occupied by several principal elements (five or more) in near-equiatomic ratios [13].

The development of HECs was stimulated by advances in high-entropy alloys [17; 18], gradually extending the concept to various ceramic applications [19]. Successful fabrication of HECs for TBCs has been reported [20–22]. Nevertheless, the synthesis route remains a bottleneck. Solid-state synthesis is the most widespread method [23–25], yet it is costly and energy-intensive due to prolonged high-energy mechanical activation and extended holding times at high temperatures for compacted powders [26]. To improve results, these operations are often repeated on the material obtained after the initial cycle [26].

Reducing HEC synthesis time is a pressing challenge in modern materials science. One proposed solution is

spark plasma sintering (SPS) [27; 28]. Nonetheless, SPS does not consistently yield single-phase HECs and may introduce extraneous contaminants that compromise the quality of the final product [29]. Recently, irradiation with intense high-energy electron beams (fast electrons) has been proposed for the synthesis of complex oxides [30]. Within a few seconds under atmospheric conditions, while maintaining almost the original bulk density of the powder mixture, various oxide ceramic materials, including phosphors [31; 32], titanates [33] and zirconolite [34], can be successfully synthesized by fast electron beam processing [35].

This study aims to synthesize high-entropy garnet-type ceramics ($Y_{0.2}Yb_{0.2}Lu_{0.2}Eu_{0.2}Er_{0.2}$)₃Al₅O₁₂ using a high-power fast electron beam.

Materials and experimental methods

Stoichiometric amounts of high-purity (99.9%) binary oxides – Y_2O_3 , Yb_2O_3 , Lu_2O_3 , Eu_2O_3 , Er_2O_3 , Al_2O_3 were dry-milled in a planetary ball mill for 30 min. The as-received oxide powders exhibited a mean particle size not exceeding 5 μm . The blended powder was subsequently loaded into a bulk copper crucible and subjected to rapid electron-beam irradiation in ambient air. The synthesis was performed on the unique scientific facility “Stand ELV-6” (Institute of Nuclear Physics SB RAS, Novosibirsk). A schematic diagram and photograph of the experimental setup are provided in Ref. [33].

The required amount of starting material was determined by calculation based on the condition of complete absorption of a 1.4 MeV electron beam within the powder layer [33], resulting in a loading density of 0.59 g/cm². Irradiation was performed in ambient air. To ensure uniform exposure, the copper crucible was translated horizontally relative to the beam axis, either with or without raster scanning across the cavity

width. Detailed descriptions of the irradiation methodology are available in Refs. [34–36]. Phase composition and morphology were analyzed post-synthesis. Scanning electron microscopy (SEM) was conducted using a Hitachi TM-3000 instrument. X-ray diffraction (XRD) patterns were recorded on an ARL X'TRA diffractometer (Switzerland). The Vickers microhardness of the solidified ceramic specimens was evaluated using a Zwick hardness testing machine (Germany).

Experimental results

Photographs of the samples before and after irradiation are shown in Fig. 1. A 1.4 MeV electron beam with a cross-sectional area of approximately 1 cm^2 on the powder surface was used for synthesis. The copper crucible was translated beneath the beam at a constant speed of $V = 1 \text{ cm/s}$. By adjusting the electron beam current, the incident power density was varied within the range of 2.8 to 16.8 kW/cm^2 .

At electron beam currents of $I_e \geq 4 \text{ mA}$, partial melting of the oxide mixture occurs, leading to the formation of spherical ceramic beads upon cooling. The mean diameter of these beads exhibits a positive correlation with the beam current. Concurrently, the fraction of unreacted feedstock powder decreases as I_e rises. Initial droplet formation is observed at the threshold current of $I_e = 4 \text{ mA}$. At $I_e = 12 \text{ mA}$, complete melting is achieved, with over 90 wt. % of the initial batch transitioning into the liquid phase. This qualitative observation is corroborated by gravimetric analysis, which compares the mass of the loaded powder before irradiation to the residual unmelted residue collected after treatment.

Prior investigations demonstrated that irradiation optimization yields high process efficiencies exceeding 90 % [37], a finding fully supported by thermodynamic modeling [38]. The solidified droplets possess a distinctly porous structure, as revealed by the cross-sectional view in Fig. 2. The material exhibits a broad pore size distribution, indicating vigorous gas evolution during melting. This phenomenon is primarily attributed to the desorption of volatile compounds physically adsorbed on the precursor powder particles.

Fig. 3 presents SEM micrographs of the teardrop-shaped ceramic bead's exterior and its fracture cross-section. The outer surface exhibits a polycrystalline morphology where individual grains display signs of coalescence across most of the area. Distinct faceting becomes apparent within surface pores; an enlarged view (Fig. 3, *a*) reveals well-defined euhedral crystals residing in these voids. Conversely, the dense regions of the fracture surface appear largely featureless at this magnification. However, high-magnification imaging of internal pores (Fig. 3, *b*, inset) confirms that the bulk volume of the ceramic possesses a developed sub-granular crystalline structure beneath the vitrified layer.

Elemental distribution maps (EDS) for all constituent cations (Al, Er, Eu, Lu, Y, Yb, O), acquired from the outer surface at high magnification (Fig. 4), indicate a homogeneous mixing of species with no observable phase separation. To verify bulk homogeneity, spot analysis was performed on the fracture cross-section. A representative spectrum of a single measurement point within the ceramic matrix (Fig. 4) reveals an elemental composition consistent with the target stoichiometry. This agreement between surface map-

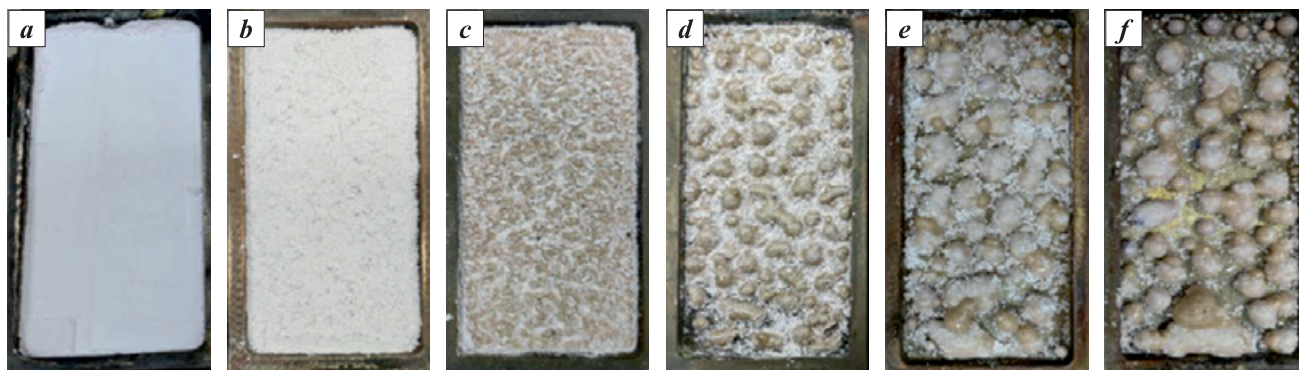


Fig. 1. Photographs of the cuvette containing the powder mixture before ($I_e = 0 \text{ mA}$) and after irradiation with 1.4 MeV fast electrons at different electron-beam currents

The cuvette travel speed beneath the beam was $V = 1 \text{ cm/s}$
 I_e , mA: *a* – 0, *b* – 2, *c* – 4, *d* – 7, *e* – 10, *f* – 12

Рис. 1. Фото кюветы с порошковой смесью до и после обработки быстрыми электронами с энергией $E = 1,4 \text{ МэВ}$ при различных значениях тока электронного пучка

Скорость перемещения кюветы под пучком $V = 1 \text{ см/с}$
 I_e , мА: *a* – 0, *b* – 2, *c* – 4, *d* – 7, *e* – 10, *f* – 12



Fig. 2. Photograph of the droplet-shaped $(Y_{0.2}Yb_{0.2}Lu_{0.2}Eu_{0.2}Er_{0.2})_3Al_5O_{12}$ ceramic product (I) showing a cross-sectional fracture (2)

The initial oxide powder mixture, Y_2O_3 , Yb_2O_3 , Lu_2O_3 , Eu_2O_3 , Er_2O_3 , Al_2O_3 was processed with a high-power fast-electron beam at $E = 1.4$ MeV, $I_e = 12$ mA, $V = 1$ cm/s

Рис. 2. Фотографическое изображение керамического продукта $(Y_{0.2}Yb_{0.2}Lu_{0.2}Eu_{0.2}Er_{0.2})_3Al_5O_{12}$ каплевой формы (I) с поперечным сколом (2)

Электронно-лучевая обработка порошковой смеси исходных оксидов Y_2O_3 , Yb_2O_3 , Lu_2O_3 , Eu_2O_3 , Er_2O_3 , Al_2O_3 проводилась в мощном пучке быстрых электронов при $E = 1,4$ МэВ, $I_e = 12$ мА, $V = 1$ см/с

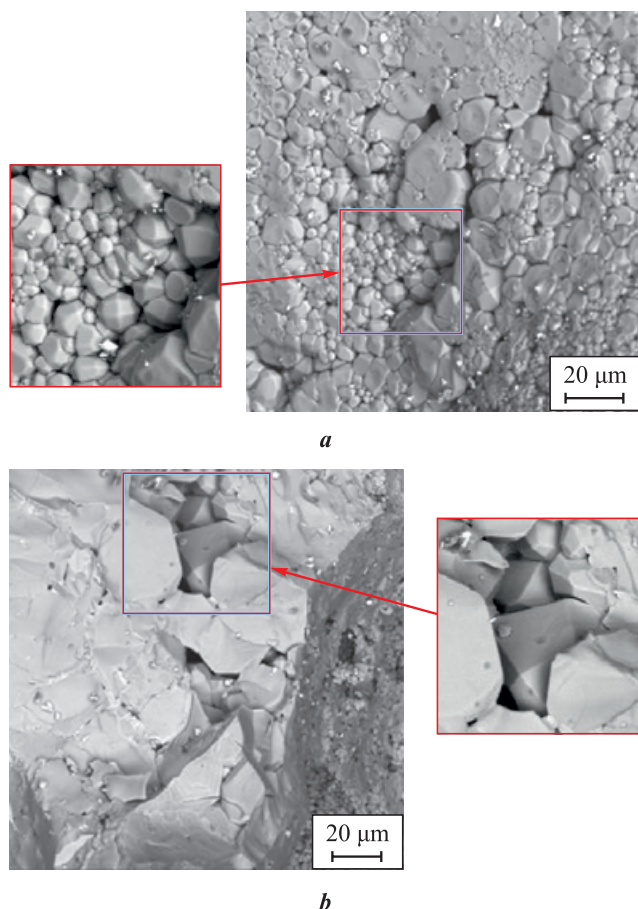


Fig. 3. SEM images of the outer surface of the droplet-shaped ceramic product (a) and its cross-sectional fracture surface (b)

Рис. 3. СЭМ-изображения внешней поверхности керамического продукта каплевой формы (a) и поверхности его поперечного скола (b)

ping and bulk probing confirms the uniform distribution of high-entropy components throughout the synthesized ceramic volume.

The X-ray diffraction patterns for the initial powder mixture, the drop-shaped ceramic product, and the powder remaining in the cuvette around it are shown in Fig. 5. The results of X-ray phase analysis of these materials are as follows: the X-ray diffraction pattern of the initial powder mixture demonstrates a superposition of peaks from the oxides included in it (Y_2O_3 , Yb_2O_3 , Er_2O_3 , Eu_2O_3 , Lu_2O_3 , Al_2O_3); the powder located outside the drop-shaped ceramic product is a mixture of an intermediate product in the form of garnets $Er_3Al_5O_{12}$, $Y_3Al_5O_{12}$ and oxides Er_2O_3 , Yb_2O_3 , Y_2O_3 , Eu_2O_3 , Lu_2O_3 , Al_2O_3 ; the drop-shaped ceramic product is a high-entropy ceramic material of the composition $(Y_{0.2}Yb_{0.2}Lu_{0.2}Eu_{0.2}Er_{0.2})_3Al_5O_{12}$ (see Fig. 5).

To evaluate the mechanical properties, microhardness measurements were conducted on the fracture cross-sections of the solidified droplets. Specimens were cold-mounted in VariDur 10 acrylic resin (Buehler, Germany) and cured into cylindrical blocks. The resulting mounts were sequentially wet-ground on a Buehler

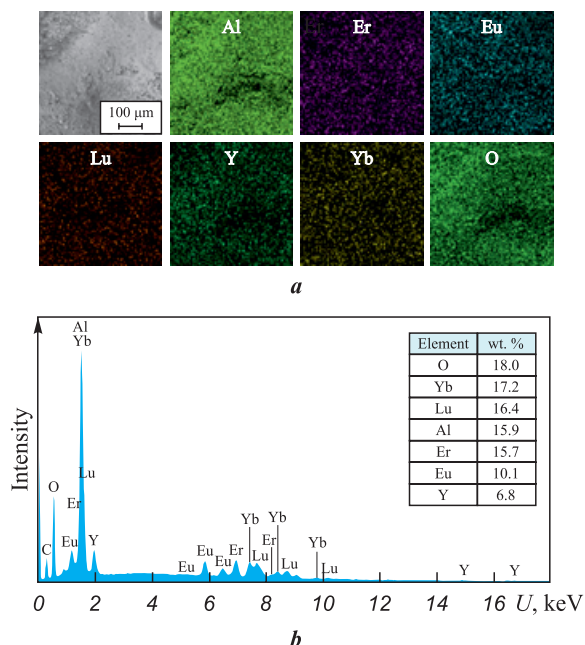


Fig. 4. SEM image of the surface of the synthesized droplet-shaped high-entropy $(Y_{0.2}Yb_{0.2}Lu_{0.2}Eu_{0.2}Er_{0.2})_3Al_5O_{12}$ ceramic product and the corresponding EDS elemental maps (a), and an EDS spectrum acquired at a selected point on the product (b)

Рис. 4. СЭМ-изображение поверхности керамической капли синтезированного высокоэнтропийного керамического продукта $(Y_{0.2}Yb_{0.2}Lu_{0.2}Eu_{0.2}Er_{0.2})_3Al_5O_{12}$ и соответствующие элементные карты ЭДС (a), а также анализ рентгенофлуоресцентного спектра в отдельной точке продукта (b)

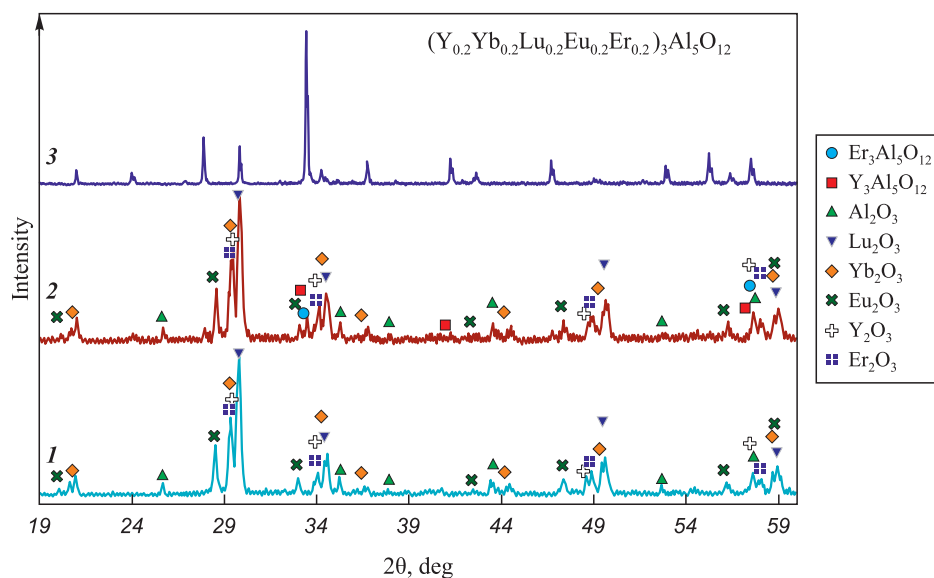


Fig. 5. XRD patterns of the initial powder mixture (1), the intermediate product remaining in the copper cuvette (2) and not involved in the formation of the droplet-shaped ceramic product, and the droplet-shaped ceramic product formed under high-power fast-electron beam irradiation (3)

Рис. 5. Дифрактограммы для исходной порошковой смеси (1), промежуточного продукта в медной чашке (2), не участвовавшего в образовании капельного керамического продукта, и материала капельного керамического продукта, образованного под воздействием мощного пучка быстрых электронов (3)

Phoenix Alpha polishing system until the geometric center of the bead was exposed. Final polishing to a mirror finish was achieved using ASM 0.3/0 HOM diamond suspension. Vickers indentations ($HV_{0.1}$) were applied exclusively to fully dense regions devoid of pores. An average microhardness value of 12.67 GPa was recorded. This high hardness, consistent with the single-phase XRD profile, confirms the successful consolidation of the $(Y_{0.2}Yb_{0.2}Lu_{0.2}Eu_{0.2}Er_{0.2})_3Al_5O_{12}$ high-entropy garnet phase.

Conclusion

This study demonstrates a novel method for synthesizing high-entropy ceramic $(Y_{0.2}Yb_{0.2}Lu_{0.2}Eu_{0.2}Er_{0.2})_3Al_5O_{12}$, a promising candidate for thermal barrier coatings. Unlike conventional furnace sintering which requires inert atmospheres or vacuum due to equipment constraints, our approach utilizes a powerful 1.4 MeV electron beam to achieve direct volumetric melting of the initial oxides in ambient air within seconds. This liquid-phase synthesis route ensures rapid homogenization of constituent elements throughout the melt pool with minimal thermal losses to the tooling.

SEM imaging coupled with EDS mapping confirms that upon rapid quenching, the drop-shaped product predominantly consists of a uniform single-phase high-entropy garnet. Minor impurities are limited to inter-

mediate garnet phases ($Er_3Al_5O_{12}$, $Y_3Al_5O_{12}$) found exclusively in the peripheral unmelted residue. XRD analysis validates the phase purity of the main droplet body. The process is characterized by intense gas evolution – likely driven by desorption of atmospheric gases, residual moisture, and released oxygen – resulting in a highly porous microstructure.

The synthesized ceramics exhibit high microhardness (12.67 GPa), confirming their dense internal structure despite surface porosity. These findings suggest significant potential for scaling this technique; however, the current formation of large monolithic droplets necessitates future development of integrated jet-milling or atomization stages to produce fine HEC powders suitable for ion-plasma deposition targets.

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V. A. Boltueva – performed XRD analysis and electron microscopy, and participated in the discussion of the results.

V. P. Krivobokov – conducted the experiments wrote the manuscript.

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