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S.A. Ghyngazov*, V.A. Boltueva, I.P. Vasil'ev

National Research Tomsk Polytechnic University, Tomsk, Russian Federation
*(*Corresponding author's e -mail: ghyngazov@tpu.ru)*

Synthesis of oxide ceramics in a beam of fast electrons

The development of technology for obtaining high-entropy ceramic materials opens up new possibilities for obtaining new heat-shielding materials. In this work, such a material is synthesized using aluminum-yttrium garnet. The effect of rapid synthesis of high-entropy ceramics within a few seconds was achieved by using an unconventional method of heating the initial mixture of powder reagents with a powerful beam of high-energy electrons. The initial mixture of Y_2O_3, Yb_2O_3, Lu_2O_3, Eu_2O_3, Er_2O_3, Al_2O_3 oxides in a stoichiometric ratio was subjected to a short-term action of a powerful beam of fast electrons under atmospheric conditions. During the radiation exposure, the powder mixture underwent melting, which led to the synthesis of high-entropy ceramics $(Y_{0.2}Yb_{0.2}Lu_{0.2}Eu_{0.2}Er_{0.2})_3Al_5O_{12}$. It was found that the melt has the form of drops with a large number of pores. The efficiency of mixture melting depends mainly on the irradiation modes and to a lesser extent on the modes of preliminary mechanical treatment of the mixture. A necessary condition for synthesis is melting of the powder mixture.

Keywords: high-entropy ceramics, thermal barrier coatings, synthesis, electron beams.

Introduction

Ceramics are the most promising material for creating thermal barrier coatings (TBC). Such outstanding parameters as high melting point, high mechanical strength, resistance to aggressive environments, the ability to vary the coefficient of linear expansion depending on the composition most fully meet the requirements for TBC. The use of simple oxides as TBC is limited due to their high thermal conductivity, the presence of phase transformations and a tendency to destruction at high temperatures. Aluminum-ytterbium garnet ($Yb_3Al_5O_{12}$) is free from the listed disadvantages inherent in simple oxides. This material is a promising candidate for their replacement. However, it is not without disadvantages such as a relatively low coefficient of thermal expansion and high thermal conductivity. One of the ways to improve these characteristics is to create a high-entropy ceramic (HEC) material based on aluminum-ytterbium garnet. At present, the HEC of the composition $(Y_{0.2}Yb_{0.2}Lu_{0.2}Eu_{0.2}Er_{0.2})_3Al_5O_{12}$ is considered as a material capable of improving the properties of $Yb_3Al_5O_{12}$. In [1, 2], it is shown that this HEC is promising for obtaining TBC. The characteristics of HECs can be varied over a wide range by selecting their composition. The emergence of HECs is largely due to the successful development of the technology for obtaining high-entropy alloys [3, 4]. Recently, a large number of types of HECs for various purposes have been created. For example, in [5], the production of $(Ti_{0.2}V_{0.2}Nb_{0.2}Mo_{0.2}W_{0.2})Si$ with high hardness (11.8 ± 0.4 GPa) and elastic modulus (387.2 ± 46.8 GPa) is reported. This material demonstrated excellent wear resistance compared to traditional single-phase ceramics. In [6], it is reported about the creation of a high-temperature ceramic of the composition $(Yb_{0.2}Tm_{0.2}Lu_{0.2}Sc_{0.2}Gd_{0.2})_2Si_2O_7$ for TBC. This material exhibits a surprisingly low thermal conductivity (1.146 W m^{-1} K^{-1} at 1100 ℃), which is even about 45.85 % lower than that of the most widely used YSZ TBC material at 1000 ℃. Seven types of thermal barrier coating materials (high-entropy rare earth tantalates $5RETaO_4$, RE= Nd, Sm, Eu, Gd, Dy, Ho, Y, Eu, Tm, Y) were synthesized in [7]. It was also shown there that $(Nd_{0.2}Dy_{0.2}Ho_{0.2}Y_{0.2}Eu_{0.2})TaO_4$ ceramics have low thermal conductivity and a suitable coefficient of thermal expansion. The methods for synthesizing HECs are diverse. In [5], HEC was obtained by spark plasma sintering, and in [6, 7], by solid-phase synthesis. In [8], HEC $(La_{0.2}Nd_{0.2}Sm_{0.2}Eu_{0.2}Gd_{0.2})_2Zr_2O_7$ was obtained by combining combustion synthesis and sintering at ultra-high pressure. In [9], zirconate rare earth ceramic powders with high entropy and low thermal conductivity were obtained by a one-pot synthesis method. In [10], HEC $(La_{1/7}Nd_{1/7}Sm_{1/7}Eu_{1/7}Gd_{1/7}Dy_{1/7}Ho_{1/7})_2Zr_2O_7$ was obtained by reactive spark plasma sintering. In [11], a dense oxide HEC $(La_{0.2}Y_{0.2}Sm_{0.2}Eu_{0.2}Gd_{0.2})_2Zr_2O_7$ with a relative density of 93.7 % was

synthesized using the cold isostatic pressing method in combination with the pressureless sintering method. This ceramic is intended for the creation of TBC.

All known methods of HECs synthesis are characterized by high temperatures, durations and their implementation requires special efforts to obtain a given phase composition of the resulting material. In this regard, a new method based on the treatment of a powder mixture of the initial components with a powerful beam of fast electrons (PBFE) is of great interest for the synthesis of HEC. The high efficiency of this method is confirmed by data on the synthesis of phosphors [12–14], zirconium corundum [15]. A feature of this method is the radiation heating of the powder mixture of the initial oxides to the melting temperature with simultaneous ionization of the mixture components by high-energy (more than 1 MeV) electrons. In this case, the synthesis of ceramics in the liquid phase occurs at an enormous speed. Ionization contributes to the acceleration of synthesis. Its stimulating effect on the synthesis of a number of ceramic materials has been convincingly proven in [11, 16–20]. To date, it has been established that synthesis under the influence of PBFE occurs in a short time with high efficiency, provided that the melting temperature of at least one of the components of the initial powder mixture is reached [15]. At the same time, the influence of sample preparation, in particular the modes of preliminary mechanical treatment, the parameters of the electron beam, especially its energy, is practically not presented in the literature.

In this work, the synthesis of HEC $(Y_{0.2}Yb_{0.2}Lu_{0.2}Eu_{0.2}Er_{0.2})_3Al_5O_{12}$ was carried out by heating the initial powder mixture with a PBFE. The regularities of melt formation depending on the conditions of mechanical treatment of the initial powder mixture of oxides and the parameters of the electron beam during irradiation and the influence of all the listed factors on the efficiency of melt formation were established.

Materials and experimental methods

Commercial powders of Y_2O_3, Yb_2O_3, Lu_2O_3, Eu_2O_3, Er_2O_3, Al_2O_3 (HK MOS-INTERNATIONAL COMPANY LIMITED, China), the purity of which was 99.9 %, were taken in accordance with the stoichiometric ratio: $3Y_2O_3 + 3Yb_2O_3 + 3Lu_2O_3 + 3Eu_2O_3 + 3\ Er_2O_3 + 25Al_2O_3 = 10(Y_{0.2}Yb_{0.2}Lu_{0.2}Eu_{0.2}Er_{0.2})_3Al_5O_{12}$. The powders were thoroughly mixed by the wet method in a planetary mill. Two processing modes were used — 500 rpm, mixing time 30 minutes (mixture 1) and 300 rpm, mixing time 90 minutes (mixture 2). The dried powder mixture was poured into the volume of a massive copper cuvette and exposed to PBFE. The value of the mass thickness was selected depending on the magnitude of the accelerating voltage, based on the condition of complete absorption of electrons in the volume of the powder mixture. Electron processing was carried out on an electron accelerator (Unique scientific installation "ELV-6 Stand", INP SB RAS, Novosibirsk) at an accelerating voltage of 1.4 MeV, 2 MeV and 2.5 MeV. The efficiency of synthesis was determined by the ratio of the mass of the synthesized ceramic product to the mass of the powder poured into the cuvette for subsequent electron processing. During irradiation, the cuvette was moved in the plane of incidence of the electron beam with the help of a movable table at speeds of 1 cm/s and 0.5 cm/s. In all cases, the beam was scanned at a frequency of 50 Hz across the width of the recess in the cuvette. Scanning electron microscopy (SEM) was carried out on a TESCAN VEGA 3 SBU electron microscope (TESCAN, Czech Republic) equipped with an OXFORD X-Max 50 attachment for X-ray fluorescence energy dispersive analysis (EDS) with a Si/Li crystal detector. X-ray phase analysis (XRD) of the initial powder mixture and the synthesized ceramic product was carried out on an X'TRA X-ray diffractometer (ARL, Switzerland).

Results and Discussion

Figure 1 shows photographs of the powder mixture of the initial oxides before processing (Fig. 1 a) and after processing with PBFE with an energy of U=1.4 MeV (Fig. 1 b), 2 MeV (Fig. 1 c) and 2.5 MeV (Fig. 1 d).

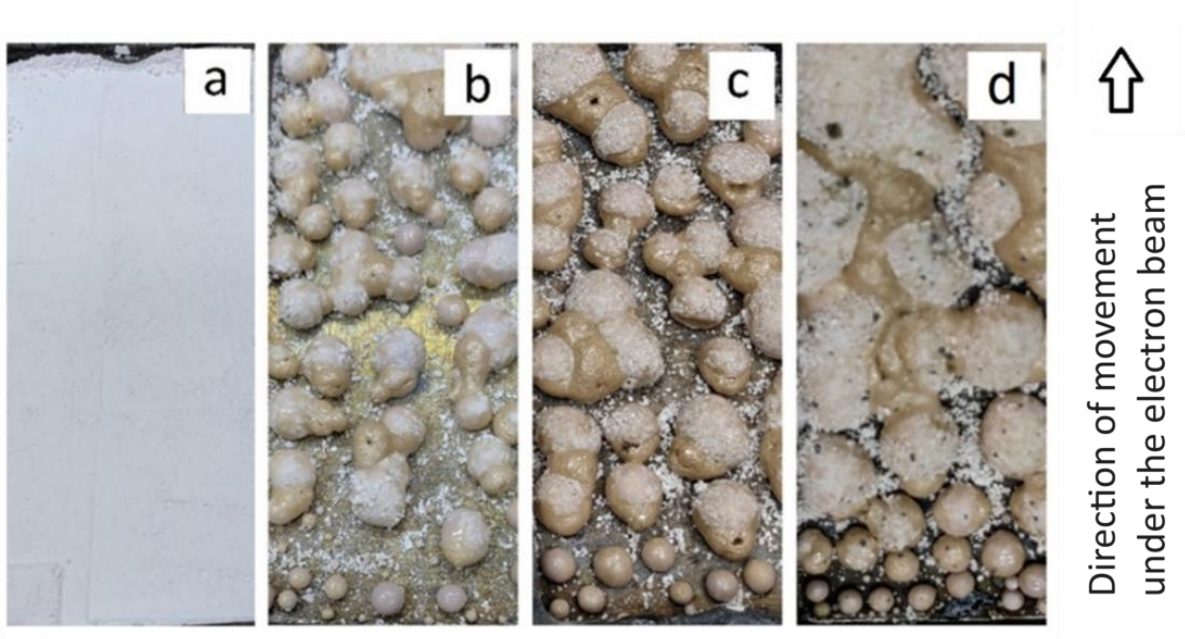

Figure 1. Effect of beam energy on melting results:
a — initial powder mixture: b — U=1.4 MeV; c — U=2.0 MeV; d — U=2.5 MeV. Beam current I=12 mA

It is evident from Figure 1 that as the accelerating voltage increases, the size of the droplets of the synthesized HEC $(Y_{0.2}Yb_{0.2}Lu_{0.2}Eu_{0.2}Er_{0.2})_3Al_5O_{12}$ increases. The characteristic decrease in the droplet size in each case as they move under the beam is explained by the end effect of energy accumulation. Regardless of the value of U, the droplets are a highly porous product (Fig. 2). The pore size increases as U increases.

Figure 2. Optical images of transverse cleavages of droplets of synthesized HEC $(Y_{0.2}Yb_{0.2}Lu_{0.2}Eu_{0.2}Er_{0.2})_3Al_5O_{12}$ at different values of accelerating voltage

The influence of mechanical treatment of the powder mixture of the initial oxides and the PBFE parameters, as well as the speed of movement of the cuvette with the powder mixture under the beam, are illustrated by the optical images of the synthesized HEC, presented in Figure 3. The PBFE parameters provided the same amount of input power. From the examination of Figure 3 it is evident that mechanical treatment affects the average size of the HEC droplet product. In this case, droplet formation strongly depends on the speed of movement of the cuvette with the powder mixture under the beam. The amount of powder product that did not participate in the formation of ceramic droplets, i.e. the efficiency of synthesis, depends on these same parameters.

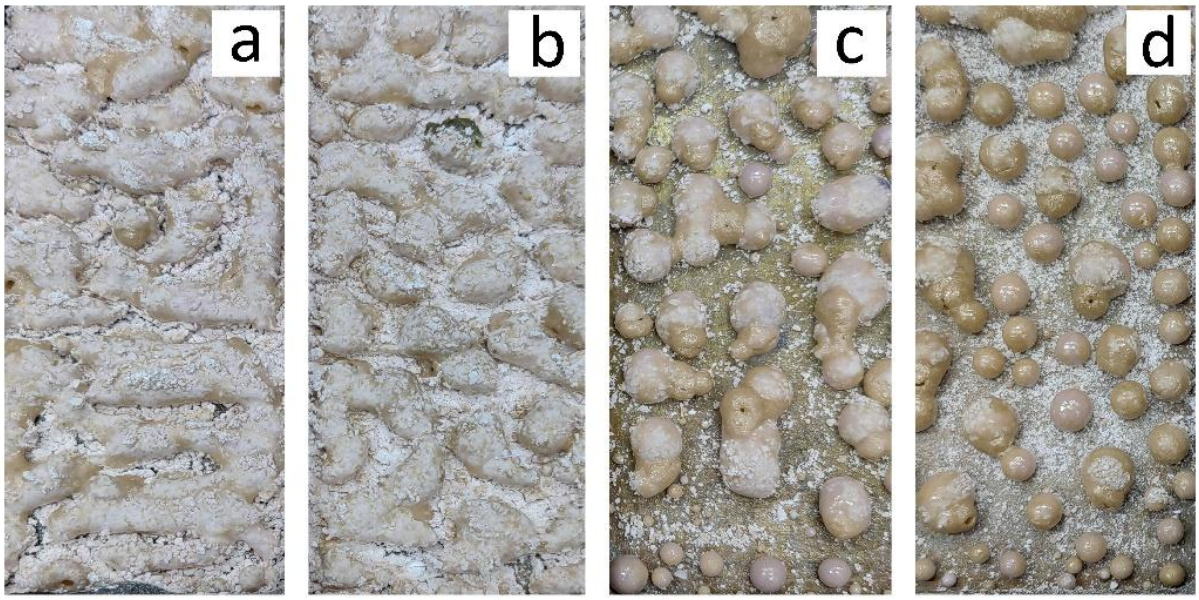

Figure 3. The influence of electron beam parameters (a, b — U=2 MeV, I=4 mA, V=0.5 cm/s, c, d — U=1.4 MeV, I=12 mA, V=1.0 cm/s) and mechanical treatment (a, c — mixture 1; b, d — mixture 2) on the formation of melt droplets

According to SEM data (Fig. 4), the outer surface of the ceramic droplets has a structured appearance with a small number of through pores (Fig. 4 a). The surface of the internal large pores in the volume of the droplet has a layered appearance with clearly defined boundaries (Fig. 4 b). On the transverse cleavage of the droplet, the crystalline structure is not traced in the interpore space, but the structure itself has a characteristic block appearance (Fig. 4 c).

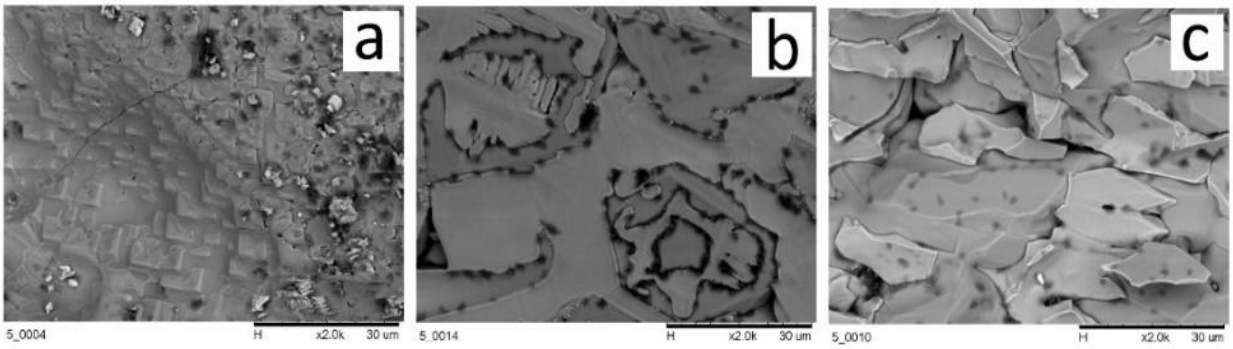

Figure 4. SEM image of a drop of synthesized HEC (U=2 MeV, I=12 mA): a — outer surface; b — inner pore surface; c — transverse cleavage surface

Weight measurements showed that the melt formation efficiency weakly depends on the conditions of mechanical processing of the initial powder mixture of oxides. At the same time, the efficiency significantly depends on the value of U and the electron beam current (Fig. 5). According to the data in Figure 6, the highest melting efficiency is achieved at U = 2 MeV.

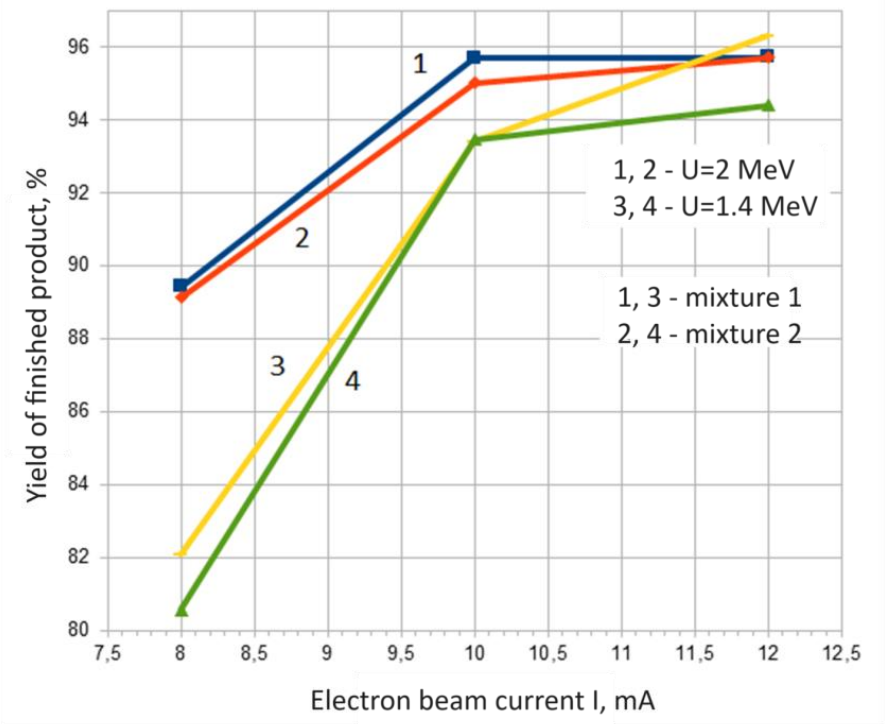

Figure 5. Dependences of the melting efficiency of mixture 1 and mixture 2 at an accelerating beam voltage of 1.4 MeV and 2 MeV on the beam current

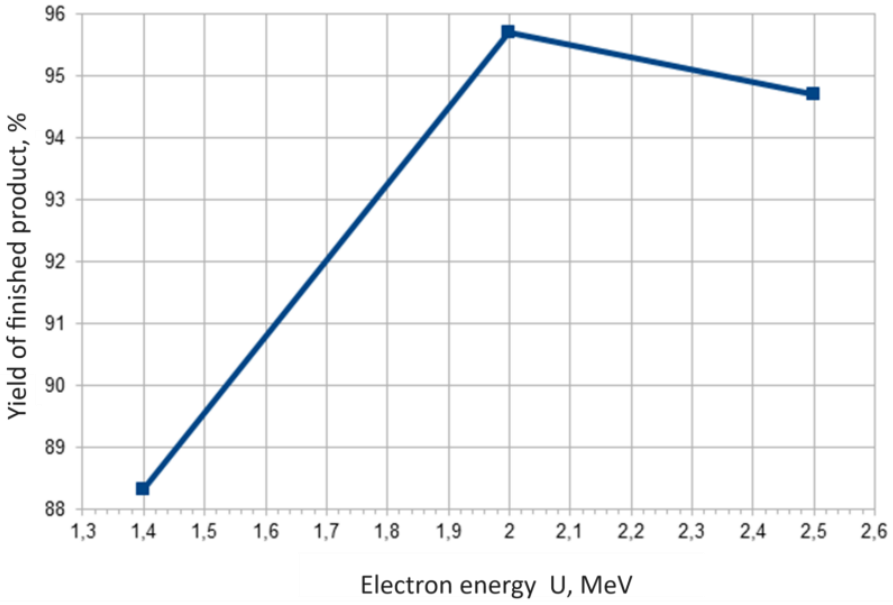

Figure 6. Effect of electron energy on the melting efficiency of powder mixture 2 at a beam current of 12 mA and a feed speed of 1 cm/s

Regardless of the mechanical processing conditions of the initial powder mixture and the PBFE processing modes, the melt droplets are HEC $(Y_{0.2}Yb_{0.2}Lu_{0.2}Eu_{0.2}Er_{0.2})_3Al_5O_{12}$. This is evidenced by the XRD data presented in Figure 7.

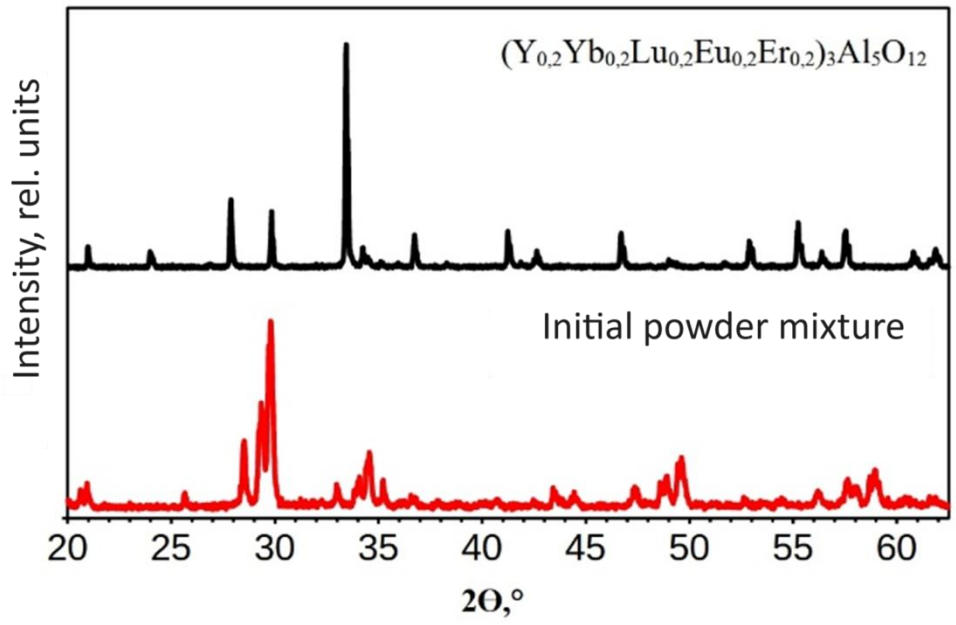

Figure 7. Diffraction patterns of the initial powder mixture of oxides and the high-entropy ceramic product $(Y_{0.2}Yb_{0.2}Lu_{0.2}Eu_{0.2}Er_{0.2})_3Al_5O_{12}$ synthesized in a powerful beam of fast electrons

Conclusions

It is shown that short-term treatment of the powder mixture of Y_2O_3, Yb_2O_3, Lu_2O_3, Eu_2O_3, Er_2O_3, Al_2O_3 oxides with a powerful beam of fast electrons leads to the synthesis of HEC $(Y_{0.2}Yb_{0.2}Lu_{0.2}Eu_{0.2}Er_{0.2})_3Al_5O_{12}$. A necessary condition for the synthesis is melting of the powder mixture. Melting leads to the formation of a highly porous ceramic product mainly in the form of drops. Preliminary mechanical treatment of the powder mixture has little effect on the melting process. The melting efficiency mainly depends on the electron energy, beam current and the speed of movement of the cuvette with the powder mixture under the beam. Regardless of the modes, the droplet ceramic product formed is high-entropy ceramics. Thus, treatment in a powerful beam of fast electrons is a new promising method for the synthesis of high-entropy ceramic materials.

Acknowledgments

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С.А. Гынгазов, В.А. Болтуева, И.П. Васильев

Жылдам электрондар шоғырындағы оксидті керамиканың синтезі

Жоғары энтропиялы керамикалық материалдарды алу технологиясының дамуы жаңа жылудан қорғайтын материалдарды алудың жаңа мүмкіндіктерін ашады. Мақалада мұндай материал алюминий-иттрий гар-торының көмегімен синтезделеді. Бірнеше секунд ішінде жоғары энтропиялы керамиканың жылдам синтезінің әсері ұнтақ реагенттерінің бастапқы қоспасын жоғары энергиялы электрондардың қуатты шоғымен қыздырудың дәстүрлі емес әдісін қолдану арқылы қол жеткізілді. Y2O3, Yb2O3, Lu2O3, Eu2O3, Er2O3, Al2O3 оксидтерінің бастапқы қоспасы стехиометриялық қатынаста атмосфералық жағдайларда жылдам электрондардың қуатты шоғының қысқа мерзімді әсеріне ұшырады. Радиациялық әсер ету кезінде ұнтақ қоспасы балқудан өтті, бұл жоғары энтропиялық керамиканың (Y0.2Yb0.2Lu0.2Eu0.2Er0.2)3Al5O12 синтезіне әкелді. Балқыманың кеуектері көп тамшылар түрінде болатыны анықталды. Қоспаны балқытудың тиімділігі негізінен сәулелену режимдеріне және аз дәрежеде қоспаны алдын ала механикалық өңдеу режимдеріне байланысты. Синтездің қажетті шарты ұнтақ қоспасын балқыту болып табылады.

Кілт сөздер: жоғары энтропиялы керамика, термиялық тосқауыл жабындары, синтез, электронды сәулелер.

С.А. Гынгазов, В.А. Болтуева, И.П. Васильев

Синтез оксидной керамики в пучке быстрых электронов

Разработка технологии получения высокоэнтропийных керамических материалов открывает новые возможности для получения новых теплозащитных материалов. В статье такой материал синтезирован с использованием алюмоиттриевого граната. Эффект быстрого синтеза высокоэнтропийной керамики в течение нескольких секунд был достигнут за счет использования нетрадиционного метода нагрева исходной смеси порошковых реагентов мощным пучком высокоэнергетических электронов. Исходная смесь оксидов Y2O3, Yb2O3, Lu2O3, Eu2O3, Er2O3, Al2O3 в стехиометрическом соотношении подвергалась кратковременному воздействию мощного пучка быстрых электронов в атмосферных условиях. В процессе радиационного воздействия порошковая смесь претерпевала плавление, что приводило к синтезу высокоэнтропийной керамики (Y0.2Yb0.2Lu0.2Eu0.2Er0.2)3Al5O12. Установлено, что расплав имеет вид капель с большим количеством пор. Эффективность плавления смеси зависит в основном от режимов облучения и в меньшей степени от режимов предварительной механической обработки смеси. Необходимым условием синтеза является плавление порошковой смеси.

Ключевые слова: высокоэнтропийная керамика, теплозащитные покрытия, синтез, электронные пучки.

Information about the authors

Ghyngazov Sergei Anatolyevich (*corresponding author*) — Doctor of technical sciences, Professor, Leading Researcher, National Research Tomsk Polytechnic University, Lenin street, 30, 634950, Tomsk, Russia; *e-mail: ghyngazov@tpu.ru*; https://orcid.org/0000-0002-2524-9238

Vasil’ev Ivan Petrovich — Candidate of technical sciences, Researcher, National Research Tomsk Polytechnic University, Lenin street, 30, 634950, Tomsk, Russia; *e-mail: zarkvon@tpu.ru*; https://orcid.org/0000-0002-4077-7012

Boltueva Valeria Aleksandrovna — Candidate of technical sciences, Junior Researcher, National Research Tomsk Polytechnic University, Lenin street, 30, 634950, Tomsk, Russia; *e-mail: kostenkova@tpu.ru*; https://orcid.org/0000-0001-8128-9042