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30.03.2026 r.

Review of the doctoral dissertation of *M.Sc. Muhammad Jahangir Khan*
Hot corrosion behaviour of a new type of thermal barrier coating materials

Received for review on February 19, 2026, this doctoral dissertation was written by Mr. Muhammad Jahangir Khan, M.Sc., at the Faculty of Materials Science and Digitalization of Industry, Silesian University of Technology, Katowice. The dissertation supervisor is Grzegorz Moskal, PhD, DSc, Eng, prof. of the SUT, and the co-supervisor is Marta Mikuśkiewicz, PhD, Eng. The PhD student's goal was to investigate and explain the mechanism of hot corrosion of thermal barrier coatings (TBC): single-phase coatings based on neodymium zirconate $\text{Nd}_2\text{Zr}_2\text{O}_7$ and more complex composite coatings such as $\text{Nd}_2\text{Zr}_2\text{O}_7 + 8\text{YSZ}$ and $\text{Nd}_2\text{Ce}_2\text{O}_7 + 8\text{YSZ}$, in environments containing liquid salt deposits of sodium and magnesium sulfates and vanadium(V) oxide.

The dissertation can be divided into several parts. In the first, theoretical part, the PhD student focused on, among other things, describing TBC systems and their importance in industry (Chapter 1), the structure of a typical barrier system, and the materials used (Chapter 2). In Section 2.4, the PhD student described TBC coating application methods, including Atmospheric Plasma Spraying, Electron Beam Physical Vapor Deposition, Suspension Plasma Spraying, and Plasma Spray Physical Vapor Deposition. He then proceeded to describe the degradation mechanisms of TBC systems, including thermal fatigue, thermal expansion coefficient mismatch, topcoat sintering and densification phenomenon, bondcoat oxidation, and high- and low-temperature hot corrosion. In subchapter 2.6, the PhD student focused on describing hot corrosion phenomena in the target materials used in his doctoral dissertation: rare-earth zirconates in two-phase systems with the addition of YSZ, using the results of studies on changes in the phase composition



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and structure of the coatings, as well as their morphology. In chapter 3, "Materials and Methods," the PhD student graphically presents his research methodology (Fig. 3.1) and then proceeds to characterize the substrate material and powder materials used to produce the TBC coatings. The PhD student utilized appropriate techniques: SEM microscopy, EDS analysis, powder X-ray diffraction, and laser diffraction to measure particle size distribution. Chapter 3 also includes a brief description of the research techniques used (PSD, XRD, SEM, EDS). The PhD student then provides a detailed description of the plasma deposition technique, process parameters, and equipment used. In subchapter 3.3, the PhD student describes the conditions for conducting high-temperature research, namely hot corrosion.

On page 42, the thesis statement is formulated and the individual research tasks are defined. The doctoral student concludes that barrier systems with a two-phase composite insulating layer composed of $\text{Nd}_2\text{Zr}_2\text{O}_7 + 8\text{YSZ}$ and $\text{Nd}_2\text{Ce}_2\text{O}_7 + 8\text{YSZ}$ will demonstrate improved resistance to hot corrosion compared to single-phase TBC systems. This is attributed to the greater ability of such composites to compensate for stresses, and therefore, to the greater crack resistance. The following chapters, chapters 5, 6, and 7, are a collection of research results that ultimately serve to demonstrate the validity of the adopted research thesis. Chapter 8 contains final conclusions that integrate the observations from the individual stages of the thesis. All chapters contain references to a total of 171 references, most of which are articles from the last two decades, and many of them are publications from the last few years. This demonstrates a solid understanding of the research problem in literature on the one hand, and on the other hand, taking into account the latest achievements in the area of production and degradation of TBC systems. In his dissertation, the doctoral student cited six references that are the result of this dissertation. In two articles (items 1 and 2, p. iii) and in chapters of two monographs (items 3 and 4, p. iii), the PhD student is the first author and corresponding author. In the article under item 5 (p. iii), the PhD student is one of the co-authors – this is a review article. The last record (item 6, p. iii) is an abstract in the book of abstracts associated with a student scientific conference. All materials are consistent with the subject of this doctoral dissertation, and the results contained in at least items 1, 2, and 5 were used in the dissertation.

Regardless of the positive opinion of the reviewed work, this dissertation may contain some ambiguities and minor errors. The PhD candidate should provide a detailed explanation of these more significant, substantive issues. However, I am merely citing minor editorial and stylistic comments and do not expect a detailed response.

General notes:



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1. The use of separate bibliography lists for each of the main chapters is not common and introduces a certain lack of coherence and continuity of thought.
2. Beginning each of Chapters 5, 6, and 7 with separate abstracts and introductions unnecessarily complicates the structure of the work and in some places even leads to duplication of information provided in Chapter 2.
3. The use of up to four reference lists also makes it difficult to navigate the doctoral dissertation.
4. The PhD student correctly used XRD, SEM, and EDS techniques in his materials research. All conclusions regarding the corrosion mechanism and the formation of specific degradation products of TBC coatings were based primarily on the results of diffraction analyses of the phase composition. Therefore, the PhD student used well-crystallized phases, which he managed to identify. This raises the question: did the PhD student verify the chemical composition (by volume or surface area) of the materials after the hot corrosion tests in any other way (besides the semiquantitative EDS technique), for example, using cross sections? In my opinion, such analyses are lacking and they would likely contribute significantly to the current interpretation.

Detailed notes:

1. This applies to the entire work. I noticed typos on almost every page, including frequent errors such as the absence of subscripts in the formulas of chemical compounds or superscripts in the symbols of ions, especially on pages 20, 27, 28, 30-32, 36, 44, 49, 55, 60-63, 80-82, and 85. It would be worthwhile to pay more attention to eliminating them in the future.
2. Figure 2.5 (p. 22) and Figure 2.7 (p. 25) are difficult to read, probably due to their low resolution.
3. Figure 2.6 (p. 23) shows points 1-16 (in red font), but the text lacks references to them and an explanation of what they mean.
4. There are numerous errors in the numbering of subsections and figures, e.g. "2.5.2 Hot Corrosion Behavior of ..." on page 24 – should be: "2.6.2 Hot corrosion...", "2.6 Summary" on page 27 – should be "2.7 Summary", "Figure 6.2" on page 69 – should be "Figure 6.3", "Figure 6.3" on page 70 – should be "Figure 6.4", "Figure 6.4" on page 71 – should be "Figure 6.5", "Figure 6.5" on page 73 – should be "Figure 6.6", "Figure 6.6" on page 74 – should be "Figure 6.7", "Figure 6.7" on page 75 – should be "Figure 6.8", "Figure 6.8" on page 76 – should be "Figure 6.9", "Figure 6.9" on page 77 – should be "Figure 6.10".



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5. Table 3.1 (p. 34). Is the given composition a nominal composition or the result of a specific measurement? A full explanation is missing.
6. Using different decimal separators, for example, a comma instead of a dot in Table 3.2 (p. 37).
7. On page 46, the sentence "Conversely, the type II type I regime prevails at higher temperatures" does not precisely indicate which regime is meant.
8. The XRD pattern in Figure 5.1 seems to indicate the presence of small amounts of ZrO_2 (at least three peaks are not indexed), while the text on page 47 states that the XRD analysis confirms the presence of pure $Nd_2Zr_2O_7$.
9. Many reaction equations were not balanced or the substrates were incorrectly written: reaction 6 (p. 76) – zirconium unbalanced, reaction 7 (p. 76) – zirconium and oxygen unbalanced, equation 3 (p. 98) – vanadium and cerium unbalanced, equation 8 (p. 49) – missing stoichiometric coefficients, equations 9 and 10 (p. 52) – erroneously balanced, and the substrate (zirconate) has a different molecular formula than that resulting from the type of TBC coating that is being tested. Reactions 11 and 12 (pp. 52 and 53) are questionable – does the NaO_2 phase actually occur here? It is possible; the diffraction pattern in Fig. 5.4 (p. 53) shows this, but there is no card marked with this symbol in the ICDD database.
10. Most of the diffraction patterns presented in the dissertation are missing units on the X-axis (2 theta). This applies in particular to: Fig. 5.6 (p. 56), Fig. 5.4 (p. 53), Fig. 6.1 (p. 66), Fig. 2.7 (p. 25), Fig. 3.2 (p. 35), Fig. 5.1 (p. 48), Fig. 5.2 (p. 49), Fig. 6.2 (p. 69), Fig. 6.5 (p. 73), Fig. 6.8 (p. 76), Fig. 7.1 (p. 86), Fig. 7.3 (p. 88), Fig. 7.6 (p. 94) and Fig. 7.9 (p. 99).
11. Fig. 7.5 (p. 92) and Fig. 7.8 (p. 97) show very interesting changes in the structure of TBC coatings caused by forced hot corrosion. Unfortunately, these figures lack descriptions that would help assess the element distribution and better understand the layered structure of the entire system (it is a pity the PhD student did not perform EDS mapping here – the polished cross section is actually predisposed to it). While a description of this layered system can be found on the previous page, the SEM images have been left alone. This is a shame because a graphical presentation of these conclusions would have improved the quality and clarity of the presented research results.
12. Figure 5.3 (p. 51). Mapping such a highly developed surface does not contribute much here – there are no visible differences in the distribution of elements.
13. Fig. 6.1 (p. 66). Not all peaks in the diffraction pattern are assigned, indicating the presence of compounds other than those expected.
14. Reference [35] on page 81 has duplicate content.



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I deliberately do not mention the remaining minor errors in this review because their impact on the quality and scientific level of the work is insignificant.

Final conclusions

To summarize the substantive content of the doctoral dissertation submitted for review, I conclude that it presents an original solution to a scientific problem. The PhD student demonstrated that the corrosion resistance of a TBC system based on $\text{Nd}_2\text{Zr}_2\text{O}_7$ in a liquid Na_2SO_4 deposit environment is satisfactory provided that the system is free of MgSO_4 and V_2O_5 . The addition of MgSO_4 results in the formation of a low-melting eutectic, and V_2O_5 reacts with $\text{Nd}_2\text{Zr}_2\text{O}_7$ to form neodymium vanadates. Both phenomena are unfavorable and contribute to the corrosion damage of the TBC coating. The $\text{Nd}_2\text{Zr}_2\text{O}_7 + 8\text{YSZ}$ composite system proposed by the PhD student demonstrated improved adhesion to the substrate during the tests. In the environment of liquid sediments, Na_2SO_4 alone and Na_2SO_4 with the addition of MgSO_4 , the pyrochloric phase of $\text{Nd}_2\text{Zr}_2\text{O}_7$ decomposed into nonstoichiometric fluorite phases. The introduction of V_2O_5 into a corrosive environment resulted in the formation of neodymium vanadate and yttrium vanadate and ZrO_2 with a monoclinic structure. The $\text{Nd}_2\text{Ce}_2\text{O}_7$ -based TBC composite system was characterized by phase stability. The PhD student did not observe decomposition of the $\text{Nd}_2\text{Ce}_2\text{O}_7$ fluorite lattice into nonstoichiometric forms. However, the presence of neodymium oxysulfates indicated the occurrence of corrosion processes. In the presence of V_2O_5 in a corrosive environment, reaction products are formed in the form of cerates depleted with neodymium vanadate and neodymium oxide with a stoichiometry similar to that of $\text{Nd}_{0.4}\text{Ce}_{0.6}\text{O}_{1.8}$.

Therefore, the dissertation by Muhammad Jahangir Khan, M.Sc., is an experimental work combining issues in materials design, material structure studies, phase transformations, and changes in the structure of TBC systems induced by high-temperature corrosion. In his research, the PhD candidate correctly applied basic research techniques and demonstrated the validity of the adopted research methodology. The results of the work have been published in journals on the JCR list. The PhD student achieved his research goals in an original way: he verified his thesis and achieved the aim of the work. I therefore declare that the doctoral dissertation by Mr. Muhammad Jahangir Khan, M.Sc., entitled "Hot corrosion behavior of a new type of thermal barrier coating materials" meets the requirements for a doctoral dissertation, as described in the Law on Higher Education and Science of July 20, 2018 (j.t. Dz. U. z 2024 r. poz. 1571) Art. 187. Therefore I request that the Scientific Discipline Council for Materials Engineering of the



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Silesian University of Technology admit Mr. Muhammad Jahangir Khan, M.Sc., to the next stages of the doctoral proceedings and to the public defense of his doctoral dissertation.

Juliusz Winiarski, D.Sc. Eng., prof. WUST



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