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### SEM STUDY OF NANO ZrC PARTICLES UNDER GAMMA IRRADIATION

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**Abstract:** This study examines the impact of gamma irradiation on nano-sized zirconium carbide (ZrC) particles using SEM and EDS analysis. SEM images at various magnifications showed no significant morphological changes or clustering after irradiation. EDS results confirmed high chemical purity, with approximately 72% Zr and 27% C, and elemental mapping revealed a uniform element distribution. The findings demonstrate that ZrC nanoparticles maintain both structural and compositional stability under gamma exposure, highlighting their potential for radiation-resistant applications.

Keywords: zirconium carbide (ZrC), gamma irradiation, SEM analysis, EDS, nanoparticle stability.

### 1. Introduction

Zirconium carbide (ZrC) is a ceramic material of significant interest in advanced materials science due to its exceptional properties, including a high melting point ( $\sim$ 3530 °C), excellent thermal conductivity, good mechanical strength, and chemical inertness. These characteristics make ZrC a promising candidate for high-temperature structural applications, particularly in aerospace, nuclear reactors, and defense technologies, where materials are exposed to extreme thermal and radiation environments. In recent years, the development of ZrC in nanoparticulate form has gained considerable attention owing to the enhanced surface area, grain boundary effects, and improved sinterability associated with nanostructured ceramics SEM is commonly used to analyze the microstructure and morphology of ZrC nanoparticles. It provides detailed images that reveal the distribution, shape, and size of the nanoparticles [1–3]. However, for applications in radiation-intensive environments—such as the core components of nuclear reactors—it is essential to understand how ionizing radiation, particularly high-energy gamma ( $\gamma$ ) rays, affects the structural integrity and compositional stability of ZrC nanoparticles.

Gamma irradiation, as a form of electromagnetic radiation with high photon energy, can induce various effects on ceramic materials, including atomic displacement, defect generation, microstructural rearrangements, and potential surface oxidation [4]. Such radiation-induced phenomena can significantly influence the physical and chemical behavior of nanoparticles, which could either enhance or deteriorate their performance in functional applications [5]. Nanomaterials are widely used in nuclear technology for both structural and functional purposes. Under high radiation conditions, these materials can develop internally ordered nano-scale structures. Such structures contribute to maintaining the mechanical strength, chemical stability, and functional properties of the materials, even under intense radiation exposure [6].

The aim of this study is to investigate the influence of gamma irradiation on the morphology and elemental composition of ZrC nanoparticles using Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDS). SEM provides high-resolution imaging to evaluate possible changes in particle size, shape, and agglomeration, while EDS enables compositional analysis and elemental mapping to assess chemical stability and homogeneity. By comparing pre- and post-irradiation samples, this study aims to provide insights into the radiation

tolerance of ZrC nanomaterials and evaluate their potential suitability for deployment in extreme operational environments.

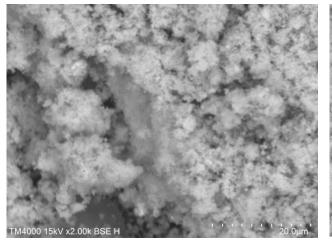
### 2. Experimental

Elemental composition analysis of ZrC nanoparticles before and after gamma irradiation was conducted using a Hitachi S-3400 scanning electron microscope (SEM) equipped with an integrated energy-dispersive X-ray spectroscopy (EDS) detector. SEM imaging was used to study the morphology and structural features of the nanoparticles, while EDS analysis provided both qualitative and quantitative data on the elemental distribution within the samples. During EDS analysis, Zirconium (Zr) and Carbon (C) were identified as the major components of the nanoparticles. Additionally, the presence of Oxygen (O) was monitored to assess potential surface oxidation caused by gamma irradiation. An increase in oxygen levels following irradiation indicated surface oxidation of the nanoparticles, highlighting the influence of gamma radiation on their surface chemistry. The results revealed significant changes in the elemental composition before and after gamma irradiation.

The oxygen content was notably higher after irradiation, suggesting the formation of an oxide layer on the surface of the ZrC nanoparticles. EDS data further provided insight into the structural stability of the nanoparticles and their elemental alterations due to gamma radiation exposure. These analyses enabled a deeper understanding of how gamma radiation influences the elemental composition and structural integrity of ZrC nanoparticles, particularly with regard to surface oxidation.

#### 3. Results and discussion

During the experiments, SEM images of the ZrC nanocomposite were acquired at various magnifications, covering both micro- and nanoscale resolutions. The primary purpose of capturing images at low magnifications in the micron range was to observe potential nanoparticle adhesion and clustering from a broader perspective. This approach is essential because if nanoparticles agglomerate to form micro-scale clusters, such features may not be detectable in high-magnification images (e.g.,  $\times 50,000$  or greater) due to their limited field of view. Therefore, as an initial step, we examine the SEM images taken before and after gamma irradiation at a magnification of  $\times 2,000$ , with a scale index of 1 µm (Figure 1).



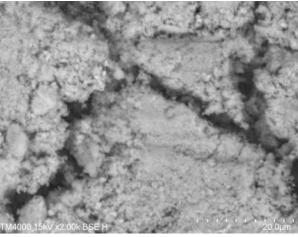
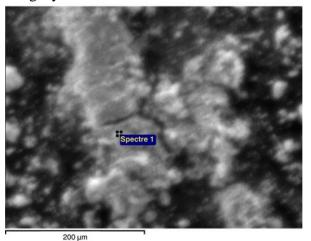


Fig. 1. SEM images of ZrC nanoparticles taken before and after gamma irradiation at a magnification of ×2,000.

The SEM images presented in Figure 1 demonstrate no significant change in the general background morphology of the sample before and after gamma irradiation. Based on these observations, it can be concluded that gamma irradiation does not induce the formation of large-scale agglomerates or microstructures in nano-ZrC. The overall morphology remains stable under the applied radiation conditions, indicating that ZrC nanoparticles retain their individual particle integrity at the microscale.



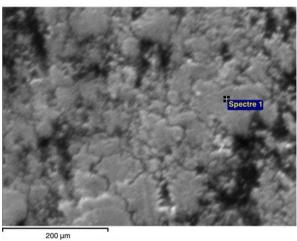


Fig. 2. SEM images of ZrC nanoparticles taken at a magnification of  $\times 5,000$ .

To assess the possibility of nanoscale clustering, SEM images were also recorded at a higher magnification of  $\times 5,000$  (with a 5  $\mu m$  scale bar), as shown in Figure 2. At this resolution, it is possible to detect relatively large nanoclusters or aggregations in the size range of approximately 200 nm to 1  $\mu m$ . However, the SEM analysis reveals no clear evidence of such clustering, confirming that the ZrC nanoparticles exhibit excellent dispersion and remain individually separated even after gamma irradiation.

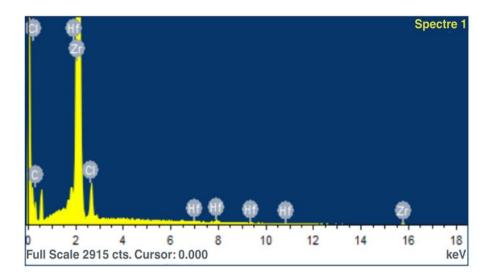


Fig. 3. Elemental composition (EDS) analysis of ZrC nanoparticles using SEM.

Elemental composition analysis was conducted using Energy Dispersive X-ray Spectroscopy (EDS) integrated into the SEM system (Figure 3). The analysis indicates that the primary constituents of the nanoparticles are zirconium (Zr) and carbon (C), with trace amounts

of chlorine (Cl) and hafnium (Hf) also detected. Quantitatively, the nanoparticles consist of approximately 72% zirconium, 27% carbon, and 1% minor elements. This composition confirms that the ZrC nanoparticles were synthesized with high chemical purity and close to the ideal stoichiometric ratio.

### 4. Conclusions

SEM analysis of nano-sized ZrC particles, both before and after gamma irradiation, revealed no significant particle coalescence or large-scale clustering at macro-, micro-, or nanoscale levels. High-resolution SEM and elemental mapping results consistently demonstrated the morphological and chemical stability of the material under gamma radiation.

EDS analysis confirmed that the synthesized ZrC nanoparticles exhibit high purity, with the zirconium-to-carbon ratio closely matching the theoretical stoichiometry of ZrC. Minor elemental traces, likely attributable to synthesis residues or surface effects, were also observed but did not compromise the structural integrity of the particles.

## References

- 1. K.B. Girish, B.N. Shobha, Synthesis and mechanical properties of zirconium nano-reinforced with aluminium alloy matrix composites, *Mater. Today Proc.*, 2018, Vol. 5, No. 1, pp. 3008–3013, <a href="https://doi.org/10.1016/j.matpr.2018.01.100">https://doi.org/10.1016/j.matpr.2018.01.100</a>
- 2. J.P. Yasnó, R.F.K. Gunnewiek, R.H.G.A. Kiminami, Microwave synthesis of ultra-high-temperature ceramic ZrC nanopowders, *Adv. Powder Technol.*, 2019, Vol. 30, No. 7, pp. 1348–1355. https://doi.org/10.1016/j.apt.2019.04.010
- 3. X. Zhu, L. Xu, C. Cui, Q. Liu, H. Wang. Preparation and characterization of zirconium carbide nanopowder by hydrothermal and carbothermal reduction methods, *Ceram. Int.*, 2024, Vol. 50, No. 17, pp. 30151–30160. https://doi.org/10.1016/j.ceramint.2024.05.314
- 4. E.M. Huseynov. FTIR spectroscopy of ZrC nanoparticles under gamma radiation. *Spectrochim. Acta A Mol. Biomol. Spectrosc.*, 2023, Vol. 286, Article ID: 122032, <a href="https://doi.org/10.1016/j.saa.2022.122032">https://doi.org/10.1016/j.saa.2022.122032</a>
- 5. S.A. Briggs, K. Hattar, Evolution of gold nanoparticles in radiation environments. In: A.T.C. Chang (Ed.), *Gold Nanoparticles Reaching New Heights*, London: IntechOpen, 2018, https://doi.org/10.5772/intechopen.80366
- 6. T.N. Agayev, V.I. Guseynov, K.T. Eyubov, S.M. Aliyev, Application of zirconium nanopowder in hydrogen energy, *J. Radiat. Res.*, 2018, Vol. 5, No. 1, pp. 12–19, Available at: <u>Issue page</u>, <u>PDF</u>

# СЭМ-ИССЛЕДОВАНИЕ НАНОЧАСТИЦ ZrC ПРИ ГАММА-ИЗЛУЧЕНИИ

### Г.И. Мурадова

**Резюме:** В данном исследовании изучается воздействие гамма-излучения на наночастицы карбида циркония (ZrC) с использованием СЭМ и ЭДС анализа. Изображения СЭМ при различных увеличениях не показали значительных морфологических изменений или кластеризации после облучения. Результаты ЭДС подтвердили высокую химическую чистоту, с приблизительно 72% Zr и 27% C, а элементное картирование показало равномерное распределение элементов. Результаты показывают, что наночастицы ZrC сохраняют

структурную и композиционную стабильность при воздействии гамма-излучения, что подчеркивает их потенциал для радиационно-устойчивых приложений.

**Ключевые слова:** карбид циркония (ZrC), гамма-излучение, СЭМ анализ, ЭДС, стабильность наночастиц

# QAMMA ŞÜALANMANIN ZrC NANOHİSSƏCİKLƏRİNƏ TƏSİRİNİN SEM İLƏ TƏDQİQİ

## G.İ. Muradova

Xülasə: Bu tədqiqatda nanoölçülü sirkonium karbid (ZrC) hissəciklərinə qamma şüalanmasının təsiri Skanedici Elektron Mikroskopiyası (SEM) və Enerji Dispersiv Rentgen Spektroskopiyası (EDS) metodları ilə araşdırılmışdır. Müxtəlif böyütmə dərəcələrində əldə edilən SEM görüntüləri şüalanmadan sonra hissəciklərin morfologiyasında və aqreqasiyasında nəzərəçarpacaq dəyişikliklərin baş vermədiyini göstərmişdir. EDS analizinin nəticələri ZrC nümunələrinin yüksək kimyəvi təmizliyə malik olduğunu (təxminən 72% Zr və 27% C) təsdiqləmiş və elementlərin sahəvi paylanmasının analizi isə onların nümunə səthi boyunca vahid paylandığını ortaya qoymuşdur. Araşdırmanın nəticələri göstərir ki, ZrC nanohissəcikləri qamma şüalanmasına məruz qaldıqda struktur və tərkib sabitliyini qoruyur, bu da onların radiasiyaya davamlı tətbiqlər üçün əlverişli material olduğunu sübut edir.

Açar sözlər: sirkonium karbid (ZrC), qamma şüalanma, SEM analizi, EDS, nanohissəciklərin stabilliyi.

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