



Original Article

EBSD studies on microstructure and crystallographic orientation of UO₂-Mo composite fuelsMurali Krishna Tummalapalli ^{a,*}, Jerzy A. Szpunar ^a, Anil Prasad ^b, Lukas Bichler ^b^a Department of Mechanical Engineering, University of Saskatchewan, Canada^b School of Engineering, University of British Columbia, Okanagan, Kelowna, Canada

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ABSTRACT

The microstructure of the fuel pellet plays an essential role in fission gas buildup and release and is critical for the safe and continued operation of nuclear power stations. Structural analysis of uranium dioxide (UO₂)-molybdenum (Mo) composite fuel pellets prepared at a range of sintering temperatures from 1300 to 1800 °C was performed. Mo micro and nanoparticles were used in making the composite pellets. A systematic investigation into the influence of processing parameters during Spark Plasma Sintering (SPS) of the pellets on the microstructure, texture, grain size, and grain boundary characters of UO₂-Mo is presented. UO₂-Mo composite show significant differences in the fraction of general boundaries and also special/coincident site lattice (CSL) boundaries. EBSD orientation maps demonstrated that <111> texturing was observed in the pellets fabricated at 1500 °C. The experimental investigations suggest that UO₂-Mo composite pellets have favorable microstructural features compared to the UO₂ pellet.

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1. Introduction

Currently (in 2021), approximately 11% of the world's total electricity is generated by nuclear reactors using uranium dioxide (UO₂) as the primary fuel source [1,2]. The UO₂ fuel can withstand harsh irradiation environments, has a high melting point, and has enhanced oxidation resistance compared to metallic fuels like nitrides or silicides [3,4]. Nevertheless, the major drawback of the present UO₂-zircalloy fuel system is the low thermal conductivity of the UO₂, which further decreases with oxidation, increasing temperatures, and irradiation damage. Therefore, the search for fissile materials with enhanced thermal conductivity is necessary. The concept of adding high thermal conductivity material into a UO₂ fuel pellet has been widely studied [3].

An essential parameter in the reactor design and safety of operation is the thermal conductivity of fuel that governs the transfer of heat produced from the nuclear fission process to making steam and electricity generation [1]. In the UO₂ fuel, the thermal conductivity degrades as the in-service temperature increases, and this creates the condition for an accident in case of loss

of coolant scenario. To reduce this possibility, composite fuel concepts were developed where the Urania fuel a metal with high thermal conductivity is added [5–8].

Molybdenum (Mo) is considered a primary candidate to be added to UO₂ due to its high thermal conductivity, high melting point, high boiling point, and low thermal neutron absorption cross-section [9]. The microstructural features of UO₂-Mo powders and pellets were examined to evaluate the impact of Mo particle size on the development of the homogeneous structure of pellets and evaluation of the porosity. The porous samples are expected to have lower thermal conductivity than high-density pellets, and therefore, high-density composite fuel pellets are more desirable for the enhancement of thermal conductivity. In contrast, microstructural defects such as micro-cracks in nuclear fuel could become a significant problem because they facilitate the release of fission gases during the normal nuclear reactor operation. Approximately 15% of inert gases such as Helium (²He), Krypton (³⁶Kr), and Xenon (⁵⁴Xe) are produced from the Uranium fission process [10]. These gasses form bubbles in the nuclear fuel and are released via open continuous porosity paths in the fuel. The resulting micro-cracks create open porosities, and a large amount of fission gas can be released through micro-cracks. Once the gas is released to free space in the fuel pin, the pressure inside the fuel pin increases, which may contribute to cladding rupture.

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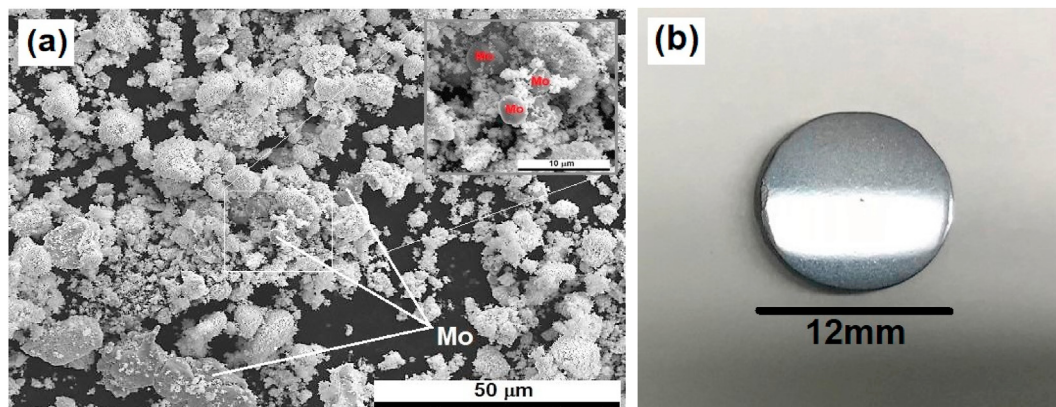


Fig. 1. (a) SEM micrograph of the UO_2 -Mo powder, (b) SPS sintered UO_2 -Mo (98 %TD) pellet of diameter 12 mm $\times$ 3 mm thickness. Sintering was done at 1800 °C, under the pressure of 60 MPa for 0.5 min.

Issues related to micro-cracking and interfacial debonding were reported for many sintered composites and are generally related to the differential coefficients of thermal expansion (CTE) of the matrix and additive particles [11–15]. Lu et al. [13] observed thermally-induced cracking in several intermetallic composites and noted that an optimum particle size exists at which the micro-cracking is suppressed. A larger particle size was seen to result in higher compressive stress in the matrix due to the lower surface-area-to-volume ratio [11]. Moreover, researchers such as Watts et al. [16] and Yeo et al. [3] noted that larger particle size can decrease the strength, hardness, and thermal conductivity of the composites.

The evolution of micro-cracks and poor interfacial contact between the UO_2 and reinforcing particles may also lead to the lowering of the thermal conductivity of the composite. Similarly, the grain boundaries are responsible for several material properties that directly influence the fuel performance, such as the fission gas build-up and release, a typical scenario of accidents related to the failure of the fuel rod [17]. Therefore, the grain boundary character of UO_2 -Mo composite pellets was analyzed using electron backscatter diffraction (EBSD). All microstructural characteristics like the grain size, texture, grain boundary character distribution, and the concentration of Σ boundaries such as ($\Sigma 3$, $\Sigma 5$, $\Sigma 9$) were examined as they influence various mechanical and kinetic properties, such as resistance to fracture, creep, and corrosion [17].

In this work, the spark plasma sintering (SPS) method was employed to fabricate UO_2 -Mo composite fuel pellets containing homogeneously dispersed Mo particles. Several studies have been pursued by various researchers on non-conventional sintering methods such as microwave sintering [18], inductive hot pressing [19], pressure-less induction heating [20], and SPS [21]. Among these sintering methods, SPS has become the most significant sintering technique for powder consolidation. Ge et al. [21] observed that by varying SPS process parameters it is possible to tune the density, grain growth, and hardness of the UO_2 pellet. The possibility of using SPS for the production of a commercial-grade fuel pellet has been investigated by Timothy et al. [22], however, an in-depth investigation of the structure and processing of composite fuel has not been reported. Thus, this work reports on a study of the influence of processing parameters on the microstructure, grain size, texture, and grain boundary characteristics of UO_2 -Mo pellets with a view of achieving a better understanding of properties of these prospective fuels.

2. Experimental

2.1. Materials and methods

A commercial uranium dioxide (UO_2) powder was provided by Cameco (Saskatoon, Canada).

Mo powder (Alfa Aesar) had a particle size of 150 μm with a purity of 99.9% (CAS: 7439-98-7).

Mo powder (Sigma-Aldrich) had a particle size of 100 nm with a purity of 99.8%.

Mo powder (Alfa Aesar) had particle sizes of 60–80 nm with a purity of 99% (CAS: 7439-98-7).

A nanometric Mo powder (US Research Nanomaterials Inc.) had particle sizes of 35–45 nm with a purity of 99.9% (CAS: 7439-98-7).

2.1.1. Starting powder

The powders of UO_2 and Mo were milled in a high-energy planetary ball mill (Torrey Hills-ND2L) in stainless steel 285 ml cups with a ball-to-powder ratio of 8:1 for 2 h, and the milling speed was maintained at 200 revolutions per minute (RPM). Iso-propanol was used as the process control agent (PCA). These powders were then characterized by energy dispersive spectroscopy (EDS) to reveal a chemical element composition. The SEM micrograph of the UO_2 -Mo powder provided in Fig. 1 indicates that the Mo particles have spongy aggregates, and the size in the range of $\sim 150 \mu\text{m}$, respectively.

2.1.2. Fabrication of UO_2 and UO_2 -Mo composites by SPS

The UO_2 -Mo powder was sintered in an argon atmosphere using a Thermal Technologies 10-3 spark plasma sintering (SPS) machine at the University of British Columbia (Kelowna, BC) Canada. The powder was placed into a graphite die with an internal diameter of 12.7 mm. A thin graphite foil (0.125 mm) was used at tool powder interfaces to prevent friction and reaction between the UO_2 -Mo powder and the graphite tool. An optical pyrometer monitored the die temperature during the experiment. The sintering temperature of pure UO_2 pellets was between 1000 °C – 1300 °C. In the case of the UO_2 -Mo-10 vol% composite, the sintering temperature had to be increased to 1300 °C – 1800 °C to achieve comparable density levels. The sintering pressure was held constant at 60 MPa and a holding time of 0.5 min at peak sintering temperature was used. After the sintering process, the pellets were ground using 500 grit silicon carbide (SiC) paper to remove the residual graphite foil for further characterization. The pellets were polished to a mirror finish by grinding them with SiC papers starting with 300 grit and

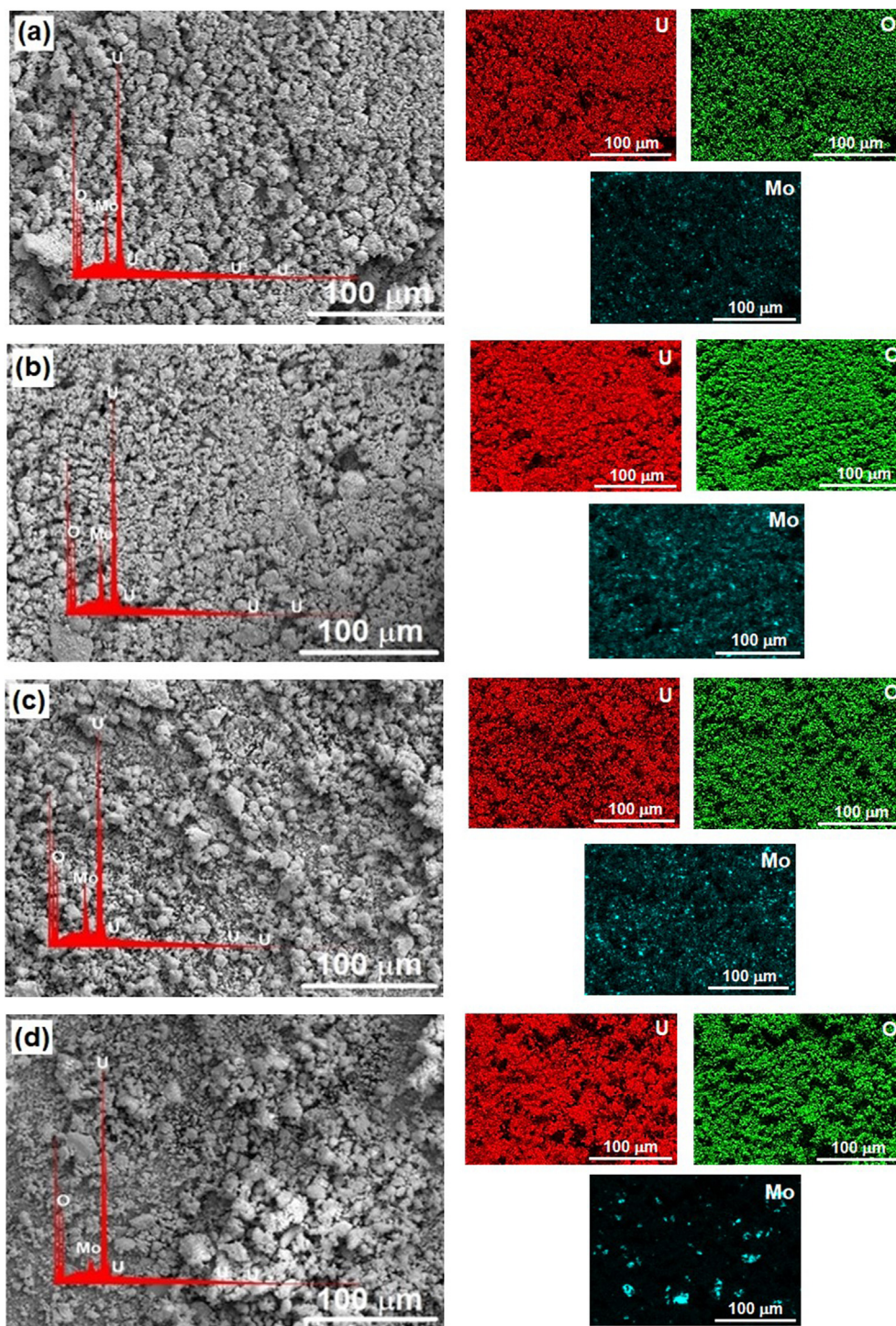


Fig. 2. Microstructure and elemental distribution of UO_2 -Mo composite. EDS spectrum and elemental mapping of UO_2 -10 vol% Mo composites with various diameters of Mo particle. (a) 35–45 nm. (b) 60–80 nm. (c) 100 nm. (d) 150 nm. Note the homogeneous distribution of Mo in the UO_2 matrix identified in small size Mo particles in a, b, c.

gradually going up to 4500 grits. After polishing with the SiC papers, the pellets were polished using 3 μm MD Mol cloth with 3 μm MD Mol suspension and using 1 μm MD Nap cloth with 1 μm MD Nap suspension. The microstructural studies on the cleaned samples were carried out using a scanning electron microscope (SEM) and electron backscatter diffraction (EBSD).

2.1.3. Scanning electron microscopy/electron backscattered diffraction

The powder and pellet samples' microstructural characterization was carried out using a Field Emission Scanning Electron Microscope (SEM) Hitachi Su 6600, equipped with an Oxford Instruments NordlysNano Electron Backscatter Diffraction (EBSD) detector and Energy Dispersive Spectrometry (EDS) detector that acquires diffraction patterns using AZTEC 2.0 data acquisition

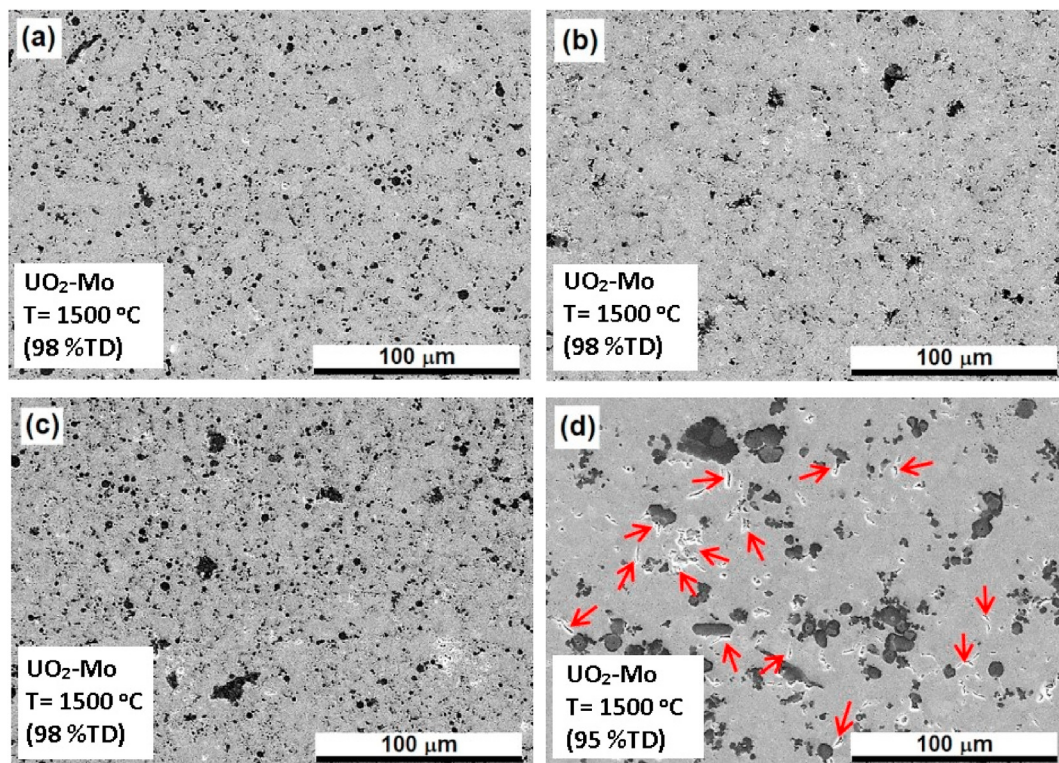


Fig. 3. Microstructure of UO_2 -10 vol% Mo composite pellets containing various sizes of Mo particle. (a) 35–45 nm. (b) 60–80 nm. (c) 100 nm. (d) 150 μm . Note cracks are originating from large size Mo particles in (d).

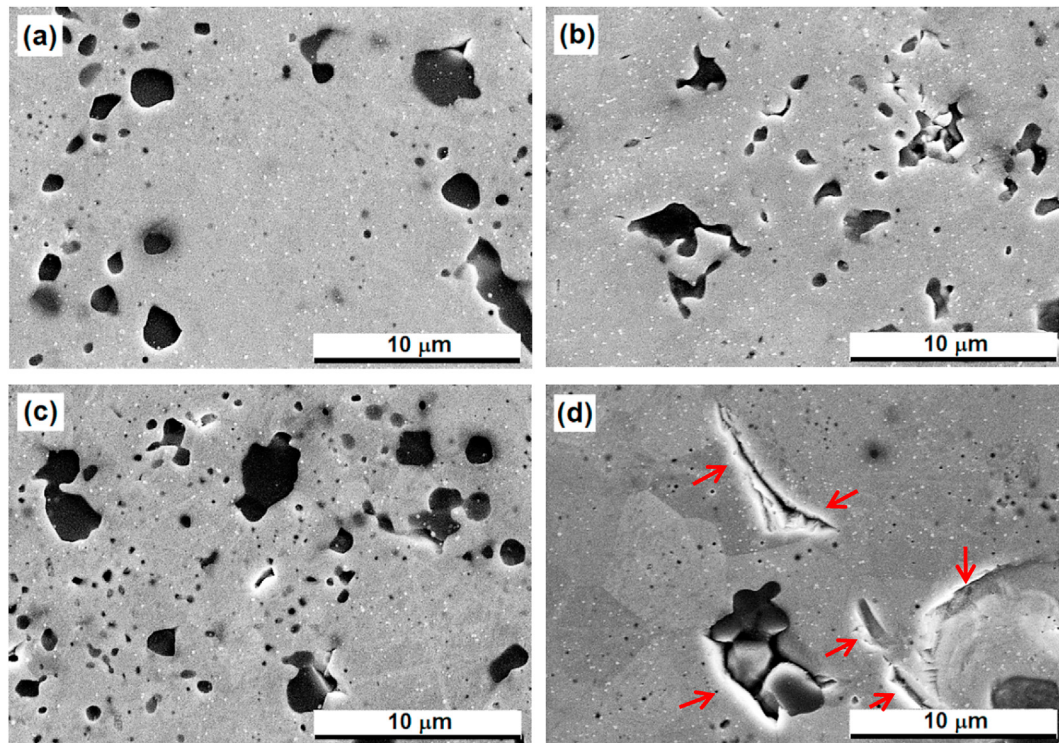


Fig. 4. Mo particles in UO_2 -10 vol% Mo composite pellets with various mean diameters. (a) 35–45 nm. (b) 60–80 nm. (c) 100 nm. (d) 150 μm . Arrows identify the cracks and debonding in the matrix and between Mo particles.

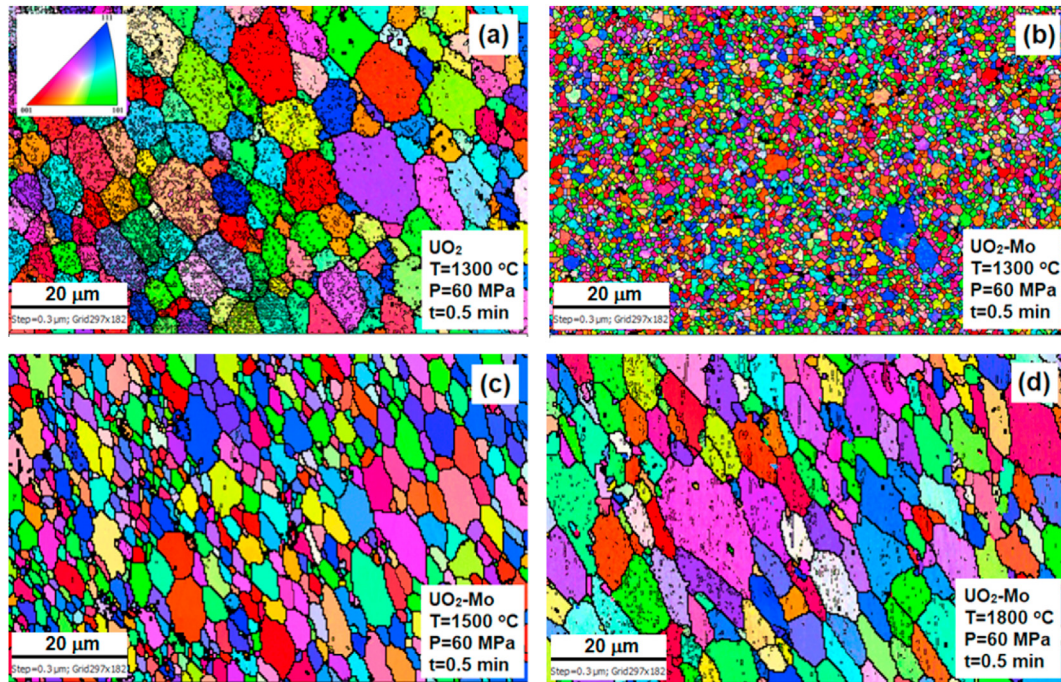


Fig. 5. (a) EBSD maps of UO_2 at 1300 °C sintering temperature with an average grain size of (5.5 μm), (b) $\text{UO}_2\text{-Mo}$ at 1300 °C sintering temperature with an average grain size of (0.9 μm), (c) $\text{UO}_2\text{-Mo}$ at 1500 °C sintering temperature with an average grain size of size (2.4 μm) (d) $\text{UO}_2\text{-Mo}$ at 1800 °C sintering temperature with an average grain size of (5.6 μm).

software. The EBSD was used to analyze the grain structure, grain boundary misorientation, coincidence site lattice-type boundaries, and texture of the specimens. The SEM and EBSD scans were measured with an acceleration voltage of 20 kV and a working distance of 9–10 mm. The post-processing of the obtained scans is completed using Channel 5 software by Oxford Instruments.

3. Results and discussions

3.1. Elemental distribution in $\text{UO}_2\text{-Mo}$ composite powder blend by EDS

The homogeneous distribution of Mo particles in the UO_2 matrix is illustrated in Fig. 2. To understand the influence of Mo particle size and distribution in the $\text{UO}_2\text{-10 vol\% Mo}$ composite powder blend, the composite was prepared with constant milling conditions and volume percentage. The 10 vol% Mo composite was prepared with different particles of sizes (i.e., the particle size of 150 μm , 100 nm, 60–80 nm, 35–45 nm) was incorporated in the UO_2 matrix via planetary ball milling. The pictures of composites with different sizes of Mo particles are shown in Fig. 2(a–d). The uniformity of Mo distribution in UO_2 for sizes 35–45 nm, 60–80 nm, 100 nm is illustrated in Fig. 2 (a, b, c). The non-homogeneous composite was noted in the large size Mo particles shown in Fig. 2 (d), respectively.

The more homogeneous distribution of small Mo particles was related due to the higher surface-area-to-volume ratio of the smaller particles. The homogeneous distribution of Mo particles influences morphology, by minimizing the porosity and reduces the visible cracks in the sintered pellets as shown in Fig. 3. Therefore, microstructural defects such as micro-cracks in fuel can be a significant problem in releasing fission gases during the nuclear reactor operation. Micro-cracks create open porosities, and a large amount of fission gas can be release through micro-cracks. Also, non-homogeneous large-size Mo particles support the hypothesis

that micro-cracking and interfacial debonding is responsible for the reduction in thermal conductivity of $\text{UO}_2\text{-Mo}$ composite [3]. In Fig. 4 (d), the 150 μm Mo particle composite has the most severe micro-cracks, and the large interfacial debonding can result in reduced thermal conductivity.

3.2. The effect of size of Mo particles on $\text{UO}_2\text{-Mo}$ composite structure

The SEM was used to analyze the micro-morphologies of the $\text{UO}_2\text{-10 vol\% Mo}$ composite sintered pellets containing the different sizes of Mo particles, namely 35–45 nm, 60–80 nm, 100 nm, and 150 μm . The different densities are also shown in Fig. 3. All the other conditions were kept constant during the sintering process. Fig. 3 shows the microstructures of these composites, where the Mo particles appear black, and the brighter area indicates the UO_2 matrix. The Mo particles seemed to have uniformity of Mo distribution in the composites as shown in Fig. 3(a–c). However, as shown in Fig. 3 (d), the composite containing 150 μm Mo particles, has cracked at the interface between a Mo particle and UO_2 matrix. The high-magnification was shown in Fig. 4 (d).

The interfaces between the UO_2 matrix and Mo particles in $\text{UO}_2\text{-10 vol\% Mo}$ composite pellets with different sizes of particles are shown in Fig. 4. The micro-cracks originating from the large-size Mo particles are presented in Fig. 4 (d), indicating that micro-cracking is less severe in composite with Mo particles of size 35–45 nm, 60–80 nm, and 100 nm compared to the composite containing 150 μm size Mo particles. There were no visible cracks in the microstructure in the composite pellets containing smaller size Mo particles. With increasing particle size, there is a more significant separation between the Mo particle and the UO_2 matrix. Fig. 4 (d) shows the interfacial debonding between UO_2 and Mo particles. The 150 μm Mo particle composite has cracks, and the large interfacial debonding can result in reduced thermal conductivity.

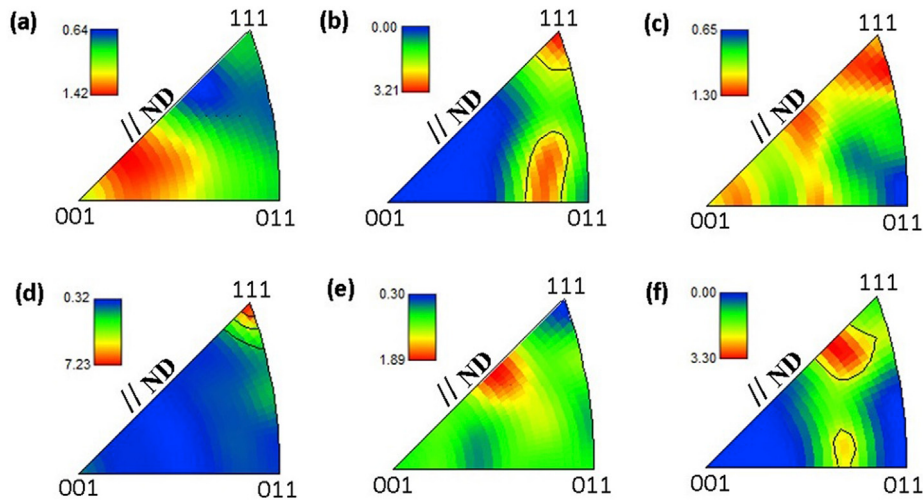


Fig. 6. Inverse pole figure of UO₂-Mo composite pellets with various sintering temperatures. (a,b) 1300 °C. (c–d) 1500 °C. (e,f) 1800 °C. Similarly, (a,c,e) shows the UO₂ phase, and (b,d,f) shows the Mo phase. (b,f) Consists of a Mo size of 35–45 nm. (d) Consists of a Mo size of 100 nm in the composite. Constant applied pressure 60 MPa and a constant sintering time of 0.5 min.

3.3. Grain size, texture, and grain boundary characterization via EBSD

3.3.1. Grain size

The mechanism of grain growth is mainly affected by the sintering temperature. Fig. 5 (a) represents the SPS UO₂ pellet at 1300 °C sintering temperature having an approximate grain size of 5.5 μm. Whereas, Fig. 5 (b, c, d) shows the orientation maps for the SPS UO₂-Mo composite as a function of sintering temperature from 1300, 1500, and 1800 °C with an average grain size of 0.9 μm, 2.4 μm, and 5.6 μm. The grain size was reduced as Mo was added because the insoluble second-phase particles were dispersed randomly in the polycrystalline solid. Therefore, the grain boundary movement during sintering was pinned by the Mo particles, and as a result, the grain size in the matrix decreased.

The comparable grain size of UO₂ and UO₂-Mo was observed at 1300 and 1800 °C sintering temperatures. This outcome is because the addition of Mo decreases the grain size of UO₂ pellets. Therefore, the higher the amount volume of secondary phase particles the more important the pinning effect, which leads to the smaller grain size. However, the sintering temperature can be increased in the UO₂-Mo composite pellets to obtain the grain size comparable with that in the UO₂ pellets.

3.3.2. Texture (Inverse pole figures)

Fig. 6 presents the comparison of the inverse pole figure (IPF) of three UO₂-Mo samples sintered at 1300 °C, 1500 °C, and 1800 °C. The results demonstrate that the grain growth activated in SPS occurred with preferential crystallographic orientation. The texture of UO₂ is rather weak oriented near <001>//ND at sintering temperature 1300 °C and moving to <111>//ND during the sintering process at 1500 °C. Similarly, at 1800 °C, the crystallographic orientation of the grains deviates from <111>//ND towards <112>//ND. The texture of Mo is oriented near <111>//ND with a major spread near <011>//ND during the sintering process at 1300 °C as shown in Fig. 6 (b). At 1500 °C there is a significant <111>//ND texture and according to the previous studies (111) texture played a significant role in enhancing the interfacial exchange energy [23] and the oxidation resistance of grains found to increase in the order of (001) < (011) < (111) [24]. Since different textures were observed, this demonstrates that the SPS sintering is a powerful tool for

producing UO₂-Mo composite pellets with a wide array of microstructural features.

3.3.3. Grain boundary character distribution (GBCD)

The grain boundaries' misorientation angle analyzed for the UO₂-Mo samples sintered at 1300 °C, 1500 °C, and 1800 °C is shown in Fig. 7. The misorientation angles are represented as a function of sintering temperature. Nerikar et al. [17] studied the UO₂ structural properties of numerous symmetrical tilt boundaries with misorientation angles ranging from 12.7° to 61.9°. They noted that the lower misorientation angles consist of edge dislocations. Whereas, for higher misorientation angles, Schottky defects (point defects in a crystal lattice) were dominant in the grain boundary structure [17].

However, for the high angle misorientation greater than 15° during the deformation; the density of dislocation will increase and the spacing between neighboring dislocations decreases; ultimately, the dislocation's cores will start to overlap, and the ordered nature of the boundary will break down. The experiments of UO₂-Mo pellets showed that there were very few high-angle boundaries in the specimens sintered at 1300 °C, 1500 °C, and 1800 °C temperatures, as shown in Fig. 7. This result reveals that SPS is, so far, a very flexible method for manufacturing the UO₂-Mo composite, with an improved structural property of the composite.

3.3.4. Coincidence site lattice (CSL) type boundaries

The presence of special/coincidence-site lattice (CSL) boundaries in UO₂ and UO₂-Mo pellet sintered at 1300 °C and 1800 °C with similar grain size is shown in Fig. 8 (a, b) and the CSL boundaries frequency distribution in the UO₂ and UO₂-Mo composite are shown in Fig. 8 (c). Based on the misorientation angle of the adjoining grains, the grain boundaries are considered as low-angle grain boundaries (LAGBs), high-angle (random) grain boundaries (HAGBs), and special/coincident-site lattice (CSL) GBs [25,26]. The different CSLs can be classified by Σ value, which represents the inverse of common lattice points in the boundaries that are formed between the two adjoining grains. Previous investigations have shown that low Σ CSL GBs, exhibited higher resistance to numerous types of intergranular degradation than HAGBs [27].

However, the high concentration of CSL GBs enhances several

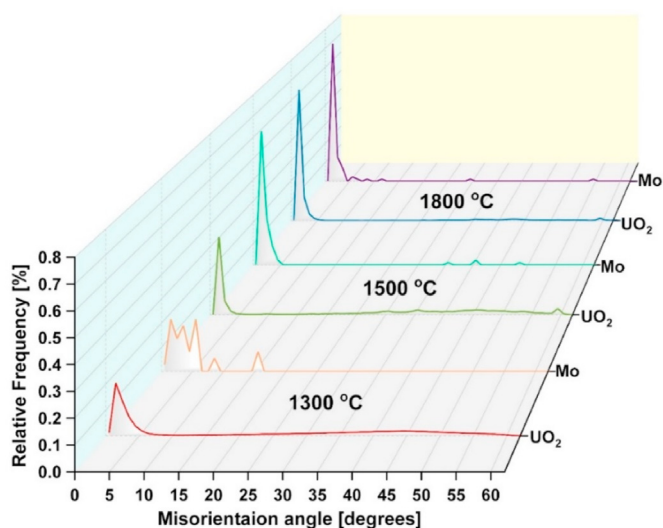


Fig. 7. Grain boundary character distribution (GBCD) of the UO_2 -Mo pellets sintered at 1300 °C, 1500 °C, and 1800 °C sintering temperature at a consistently applied pressure of 60 MPa and a constant sintering time of 0.5 min.

properties of the materials. It was demonstrated that grain boundary sliding during creep can be lowered [28,29]. Resistance to solute segregation, precipitation, and intergranular embrittlement [30] can be improved. Similarly, the resistance to high-temperature fracture [31], welding ability [32], resistance to intergranular

corrosion [33], resistance to corrosion and stress corrosion [30,34,35], and resistance to radiation damage [29,36] will be improved. The low Σ boundaries of $\Sigma 3$, $\Sigma 5$, and $\Sigma 9$ GBs shown in red, green, and pink colors. The statistical analysis of the frequency of CLS shows that there are a higher fraction of these boundaries in the UO_2 -Mo than the UO_2 pellets.

4. Summary

We have presented an extensive experimental analysis that will allow controlling microstructure, grain size, texture, grain boundary characteristics of the UO_2 -Mo pellets. It was noted that by varying the Mo particle size, it is possible to tune the microstructural characteristics of UO_2 -Mo composite fuel and the small size of particles allows very homogeneous distribution in the matrix. However, large Mo particles initiate micro-cracks and that might lower the thermal conductivity and can be a significant problem in releasing fission gases during the nuclear reactor operation. Additionally, we prepared three UO_2 -Mo pellets of varying sintering temperatures by SPS technique and the EBSD analysis demonstrated that higher sintering temperatures result in larger grain size. However, the molybdenum particles retards grain growth, and the size of grains in the composite is smaller than in pure UO_2 when sintered at the same temperature. Therefore, the sintering temperature can be increased to obtain a similar grain size to that of the UO_2 pellets. By optimizing the processing parameters, the texture of the sintered pellets can be modified. The orientation maps demonstrated that $\langle 111 \rangle$ texturing was observed in the pellets

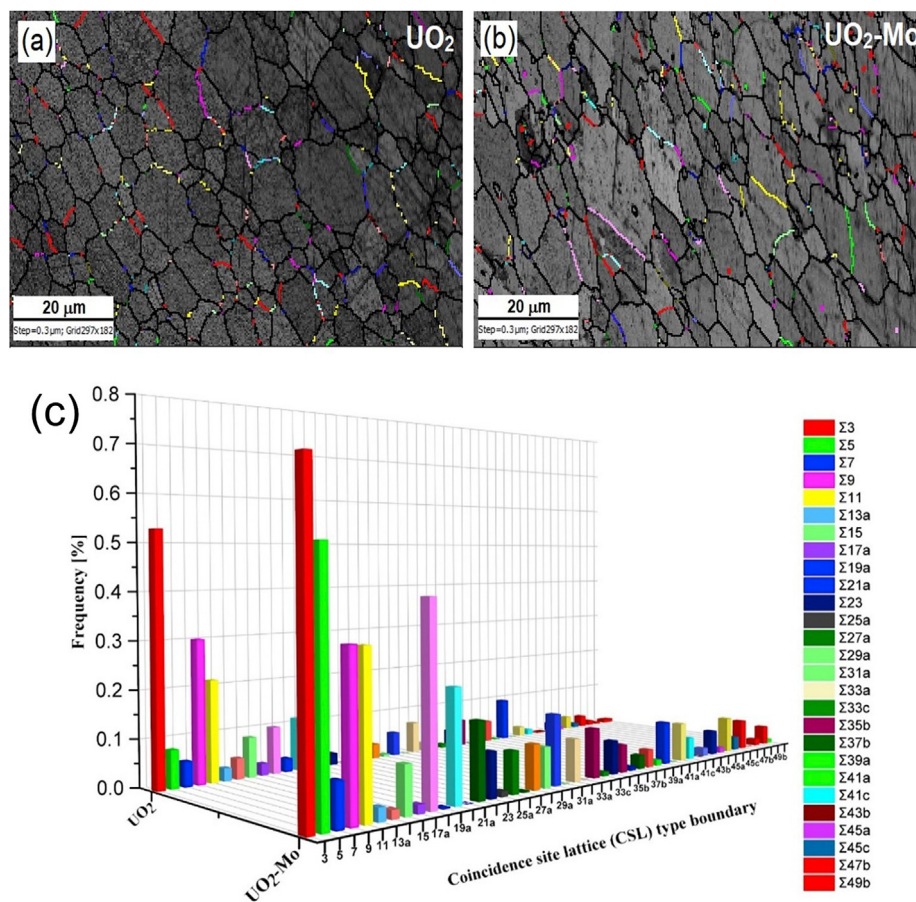


Fig. 8. EBSD map (a, b) band contrast with coincidence site lattice (CSL) boundaries. (c) CSL boundaries versus frequency of the UO_2 and UO_2 -Mo at similar grain sizes.

sintered at 1500 °C. Our studies revealed that there are very few relative frequencies of high-angle boundaries in the pellets fabricated from 1300–1800 °C temperatures, respectively. The experimental investigations also suggest that a higher percentage of CSL grain boundaries in UO₂-Mo composite pellets should be beneficial for the improvement of the resistance to failure.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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References

- [1] K. Gofryk, S. Du, C.R. Stanek, J.C. Lashley, X.Y. Liu, R.K. Schulze, J.L. Smith, D.J. Safarik, D.D. Byler, K.J. McClellan, B.P. Uberuaga, B.L. Scott, D.A. Andersson, Anisotropic thermal conductivity in uranium dioxide, *Nat. Commun.* 5 (2014) 1–7, <https://doi.org/10.1038/ncomms5551>.
- [2] M. Lenzen, Current state of development of electricity-generating technologies: a literature review, *Energies* 3 (2010) 462–591, <https://doi.org/10.3390/en3030462>.
- [3] S. Yeo, E. McKenna, R. Baney, G. Subhash, J. Tulenko, Enhanced thermal conductivity of uranium dioxide-silicon carbide composite fuel pellets prepared by Spark Plasma Sintering (SPS), *J. Nucl. Mater.* 433 (2013) 66–73, <https://doi.org/10.1016/j.jnucmat.2012.09.015>.
- [4] S.C. Finkeldei, J.O. Kiggans, R.D. Hunt, A.T. Nelson, K.A. Terrani, Fabrication of UO₂-Mo composite fuel with enhanced thermal conductivity from sol-gel feedstock, *J. Nucl. Mater.* (2019), <https://doi.org/10.1016/j.jnucmat.2019.04.011>.
- [5] W.J. Weber, A. Navrotsky, S. Stefanovsky, E.R. Vance, E. Vernaz, Materials science of high-level immobilization, *MRS Bull.* 34 (2009) 46–52.
- [6] L.J. Ott, K.R. Robb, D. Wang, Preliminary assessment of accident-tolerant fuels on LWR performance during normal operation and under DB and BDB accident conditions, *J. Nucl. Mater.* 448 (2014) 520–533, <https://doi.org/10.1016/j.jnucmat.2013.09.052>.
- [7] L.H. Ortega, B.J. Blamer, J.A. Evans, S.M. McDevitt, Development of an accident-tolerant fuel composite from uranium mononitride (UN) and uranium sesquioxide (U₃Si₂) with increased uranium loading, *J. Nucl. Mater.* 471 (2016) 116–121, <https://doi.org/10.1016/j.jnucmat.2016.01.014>.
- [8] X.M. Bai, M.R. Tonks, Y. Zhang, J.D. Hales, Multiscale modeling of thermal conductivity of high burnup structures in UO₂ fuels, *J. Nucl. Mater.* 470 (2016) 208–215, <https://doi.org/10.1016/j.jnucmat.2015.12.028>.
- [9] D.J. Kim, Y.W. Rhee, J.H. Kim, K.S. Kim, J.S. Oh, J.H. Yang, Y.H. Koo, K.W. Song, Fabrication of micro-cell UO₂-Mo pellet with enhanced thermal conductivity, *J. Nucl. Mater.* 462 (2015) 289–295, <https://doi.org/10.1016/j.jnucmat.2015.04.003>.
- [10] J. Hu, A.C. Hayes, W.B. Wilson, Rizwan-Uddin, Fission gas production in reactor fuels including the effects of ternary fission, *Nucl. Eng. Des.* 240 (2010) 3751–3757, <https://doi.org/10.1016/j.nucengdes.2010.08.020>.
- [11] R.I. Todd, B. Derby, Thermal stress induced microcracking in alumina-20% SiCp composites, *Acta Mater.* 52 (2004) 1621–1629, <https://doi.org/10.1016/j.actamat.2003.12.007>.
- [12] B.R. Powell, G.E. Youngblood, D.P.H. Hasselman, L.D. Bentsen, Effect of thermal expansion mismatch on the thermal diffusivity of glass-Ni composites, *J. Am. Ceram. Soc.* 63 (1980) 581–586, <https://doi.org/10.1111/j.1151-2916.1980.tb10769.x>.
- [13] T.C. Lu, J. Yang, Z. Suo, A.G. Evans, R. Hecht, R. Mehrabian, Matrix cracking in intermetallic composites caused by thermal expansion mismatch, *Acta Metall. Mater.* 39 (1991) 1883–1890, [https://doi.org/10.1016/0956-7151\(91\)90157-V](https://doi.org/10.1016/0956-7151(91)90157-V).
- [14] S.Y. Fu, X.Q. Feng, B. Lauke, Y.W. Mai, Effects of particle size, particle/matrix interface adhesion and particle loading on mechanical properties of particulate-polymer composites, *Compos. B Eng.* 39 (2008) 933–961, <https://doi.org/10.1016/j.compositesb.2008.01.002>.
- [15] B. Burton, G.L. Reynolds, J.P. Barnes, The influence of grain size on the creep of uranium dioxide, *J. Mater. Sci.* 8 (1973) 1690–1694, <https://doi.org/10.1007/BF02403517>.
- [16] J. Watts, G. Hilmas, W.G. Fahrenholtz, Mechanical characterization of ZrB₂-SiC composites with varying SiC particle sizes, *J. Am. Ceram. Soc.* 94 (2011) 4410–4418, <https://doi.org/10.1111/j.1551-2916.2011.04885.x>.
- [17] P.V. Nerikar, K. Rudman, T.G. Desai, D. Byler, C. Unal, K.J. McClellan, S.R. Phillpot, S.B. Sinnott, P. Peralta, B.P. Uberuaga, C.R. Stanek, Grain boundaries in uranium dioxide: scanning electron microscopy experiments and atomistic simulations, *J. Am. Ceram. Soc.* 94 (2011) 1893–1900, <https://doi.org/10.1111/j.1551-2916.2010.04295.x>.
- [18] J.H. Yang, K.W. Song, Y.W. Lee, J.H. Kim, K.W. Kang, K.S. Kim, Y.H. Jung, Microwave process for sintering of uranium dioxide, *J. Nucl. Mater.* 325 (2004) 210–216, <https://doi.org/10.1016/j.jnucmat.2003.12.003>.
- [19] I. Amato, R.L. Colombo, A.M.P. Balzari, Hot-pressing of uranium dioxide, *J. Nucl. Mater.* 20 (1966) 210–214, [https://doi.org/10.1016/0022-3115\(66\)90009-2](https://doi.org/10.1016/0022-3115(66)90009-2).
- [20] J.H. Yang, Y.W. Kim, J.H. Kim, D.J. Kim, K.W. Kang, Y.W. Rhee, K.S. Kim, K.W. Song, Pressureless rapid sintering of UO₂ assisted by high-frequency induction heating process, *J. Am. Ceram. Soc.* 91 (2008) 3202–3206, <https://doi.org/10.1111/j.1551-2916.2008.02615.x>.
- [21] L. Ge, G. Subhash, R.H. Baney, J.S. Tulenko, E. McKenna, Densification of uranium dioxide fuel pellets prepared by spark plasma sintering (SPS), *J. Nucl. Mater.* 435 (2013) 1–9, <https://doi.org/10.1016/j.jnucmat.2012.12.010>.
- [22] T. Ironman, J. Tulenko, G. Subhash, Exploration of viability of spark plasma sintering for commercial fabrication of nuclear fuel pellets, *Nucl. Technol.* 200 (2017) 144–158, <https://doi.org/10.1080/00295450.2017.1360714>.
- [23] Y.T. Chen, The effect of interface texture on exchange biasing in Ni₈₀Fe₂₀/Ir₂₀Mn₈₀ system, *Nanoscale Res. Lett.* 4 (2009) 90–93, <https://doi.org/10.1007/s11671-008-9207-4>.
- [24] X. Wang, F. Fan, J.A. Szpunar, L. Zhang, Influence of grain orientation on the incipient oxidation behavior of Haynes 230 at 900 °C, *Mater. Char.* 107 (2015) 33–42, <https://doi.org/10.1016/j.matchar.2015.06.029>.
- [25] C.B. Thomson, V. Randle, The effects of strain annealing on grain boundaries and secure triple junctions in nickel 200, *J. Mater. Sci.* 32 (1997) 1909–1914, <https://doi.org/10.1023/A:1018573327408>.
- [26] H. Akhiani, M. Nezakat, M. Sanayei, J. Szpunar, The effect of thermo-mechanical processing on grain boundary character distribution in Incoloy 800H/HT, *Mater. Sci. Eng., A* 626 (2015) 51–60, <https://doi.org/10.1016/j.msea.2014.12.046>.
- [27] T. Watanabe, Part IV Applications : grain boundary engineering and microstructural design for advanced properties, in: *Microstruct. Des. Adv. Eng. Mater.*, Wiley-VCH Verlag GmbH, Weinheim, Germany, 2013, pp. 403–446, <https://doi.org/10.1051/forest>.
- [28] S. Spigarelli, M. Cabibbo, E. Evangelista, G. Palumbo, Analysis of the creep strength of a low-carbon AISI 304 steel with low-Σ grain boundaries, *Mater. Sci. Eng., A* 352 (2003) 93–99, [https://doi.org/10.1016/S0921-5093\(02\)00903-6](https://doi.org/10.1016/S0921-5093(02)00903-6).
- [29] V. Randle, Sigma-boundary statistics by length and number, *Interface Sci.* 10 (2002) 271–277, <https://doi.org/10.1023/A:1020877528820>.
- [30] J.J. Kai, F.R. Chen, T.S. Duh, Effects of grain boundary misorientation on radiation-induced solute segregation in proton irradiated 304 stainless steels, *Mater. Trans.* 45 (2004) 40–50, <https://doi.org/10.2320/matertrans.45.40>.
- [31] L. Tan, T.R. Allen, J.T. Busby, Grain boundary engineering for structure materials of nuclear reactors, *J. Nucl. Mater.* 441 (2013) 661–666, <https://doi.org/10.1016/j.jnucmat.2013.03.050>.
- [32] L. Tan, K. Sridharan, T.R. Allen, R.K. Nanstad, D.A. McClintock, Microstructure tailoring for property improvements by grain boundary engineering, *J. Nucl. Mater.* 374 (2008) 270–280, <https://doi.org/10.1016/j.jnucmat.2007.08.015>.
- [33] S. Xia, H. Li, T.G. Liu, B.X. Zhou, Applying grain boundary engineering to Alloy 690 tube for enhancing intergranular corrosion resistance, *J. Nucl. Mater.* 416 (2011) 303–310, <https://doi.org/10.1016/j.jnucmat.2011.06.017>.
- [34] E.A. West, G.S. Was, IGSCC of grain boundary engineered 316L and 690 in supercritical water, *J. Nucl. Mater.* 392 (2009) 264–271, <https://doi.org/10.1016/j.jnucmat.2009.03.008>.
- [35] G. Palumbo, E.M. Lehigh, P. Lin, Applications for grain boundary engineered materials, *Jom* 50 (1998) 40–43, <https://doi.org/10.1007/s11837-998-0248-z>.
- [36] M. Sekine, N. Sakaguchi, M. Endo, H. Kinoshita, S. Watanabe, H. Kokawa, S. Yamashita, Y. Yano, M. Kawai, Grain boundary engineering of austenitic steel PNC316 for use in nuclear reactors, *J. Nucl. Mater.* 414 (2011) 232–236, <https://doi.org/10.1016/j.jnucmat.2011.03.049>.