

# RECENT PROGRESS IN OXIDE-DISPERSION-STRENGTHENED (ODS) ALLOYS PRODUCED BY ADDITIVE MANUFACTURING

## NAJNOVEJŠI NAPREDEK NA PODROČJU Z OKSIDI DISPERZIJSKO UTRJENIH ZLITIN, IZDELANIH Z DODAJALNIMI TEHNOLOGIJAMI

Paul McGuinness<sup>1\*</sup>, Irena Paulin<sup>1</sup>, Črtomir Donik<sup>1</sup>, Anna Dobkowska<sup>2</sup>,  
Jirí Kubásek<sup>3</sup>, Jan Pokorný<sup>3</sup>, Matjaž Godec<sup>1</sup>

<sup>1</sup>Institute of Metals and Technology, Lepi pot 11, Ljubljana, Slovenia

<sup>2</sup>Faculty of Materials Science and Engineering, Warsaw University of Technology, Warsaw, Poland

<sup>3</sup>Department of Metals and Corrosion Engineering, University of Chemistry and Technology, Prague, Czech Republic

*Prejem rokopisa – received: 2025-01-05; sprejem za objavo – accepted for publication: 2025-1-09*

doi:10.17222/mit.2025.1364

Oxide-dispersion-strengthened (ODS) alloys exhibit exceptional mechanical properties, making them ideal for high-temperature applications in areas such as aerospace and nuclear reactors. The traditional manufacturing of ODS alloys involves mechanical alloying, followed by processes such as hot extrusion and hot isostatic pressing. However, these methods are limited when it comes to producing complex geometries. Recent advances in additive manufacturing (AM) techniques, specifically selective laser melting (SLM) and directed-energy deposition (DED), offer exciting new possibilities for fabricating ODS alloys. Early research demonstrated the feasibility of using SLM to create complex parts with uniformly dispersed oxide particles, thereby enhancing the materials' properties. Subsequent studies confirmed that optimising the SLM parameters could further improve the mechanical performance of ODS alloys. DED techniques have also shown promise, with innovations like in-situ oxide formation during deposition and high-speed laser cladding. These methods have achieved success by producing ODS materials with refined microstructures and enhanced mechanical properties. The latest research continues to explore the potential of AM for ODS alloys, focusing on improving the dispersion of nanoparticles and minimising the tendency of particles to agglomerate. Overall, AM has advanced the fabrication of ODS alloys by offering efficient production routes and the ability to create intricate designs with superior properties.

**Key words:** Oxide-dispersion-strengthened alloys, additive manufacturing, recent progress and research

Zlitine ojačane z disperzijo oksidnih delcev (ODS; angl.: Oxide-Dispersion-Strengthened alloys) imajo izjemne mehanske lastnosti in so zato še posebej idealne za visoko temperaturne aplikacije v letalski industriji in jedrskih reaktorjih. Konvencionalne ODS zlitine so izdelane s P/M (angl.: Powder Metallurgy) postopkom mehanskega legiranja, kateremu sledijo postopki zgoščevanja kot sta naprimer vroča ekstruzija ali vroče izostatsko stiskanje (HIP). Vendar so ti postopki omejeni predvsem pri izdelavi majhnih izdelkov zahtevnih geometrij. Najnovejši napredek dodajalnih tehnologij (AM; angl.: additive manufacturing), še posebej selektivnega laserskega taljenja (SLM; angl.: selective laser melting) in direktnega energijskega nanosa (DED; angl.: directed-energy deposition), ponuja vznemirljive nove možnosti izdelave ODS zlitin. Nedavne raziskave so pokazale možnosti uporabe SLM za kreiranje izdelkov zahtevnih oblik z enakomerno porazdelitvijo oksidnih delcev v kovinski matrici, kar močno izboljša njihove lastnosti. Nadalje so študije potrdile, da optimiziranje parametrov SLM lahko še izboljša mehanske lastnosti ODS zlitin. Tehnike tehnologije DED so prav tako obetajoče. Z inovacijami kot sta *in-situ* tvorba oksidov med nanosom in visoko hitrostno lasersko platiranje se lahko doseže izdelava ODS kompozitnih materialov z zelo fino mikrostrukturo in odličnimi mehanskimi lastnostmi. Najnovejše raziskave se nadaljujejo na področju raziskovanja potenciala AM tehnologij za izdelavo novih vrst ODS zlitin z osredotočenjem na izboljšanje disperzije (fine enakomerne porazdelitve) nanodelcev in zmanjšanje tendence njihovega skupljanja (aglomeracije). Dodajalna tehnologija torej ponuja napredek pri izdelavi ODS zlitin in ponuja učinkovite izdelovalne postopke ter možnost kreiranja posebno zahtevnih oblik (dizajnov) oziroma izdelkov s superiornimi lastnostmi.

**Ključne besede:** zlitine ojačane s fino disperzijo oksidnih delcev, dodajalne tehnologije, najnovejše raziskave in napredek

## 1 INTRODUCTION

Oxide-dispersion-strengthened (ODS) alloys are a category of materials known for their exceptional mechanical properties at both ambient and high temperatures.<sup>1-4</sup> These characteristics make them ideal for use in gas turbines and combustion engines in aerospace,<sup>5</sup> as well as in nuclear-fission-reactor cladding tubes and nuclear-fusion reactor blankets. The remarkable properties

of ODS materials stem from the uniformly distributed sub-micrometer oxide particles within a corrosion-resistant metallic matrix. Typically, the production of ODS materials involves mechanical alloying,<sup>6</sup> followed by hot extrusion or hot isostatic pressing (HIP),<sup>7</sup> hot rolling,<sup>8</sup> and final shaping.<sup>9</sup> Although there are alternative methods to achieve the desired microstructures, they are infrequently documented. Some of these include gas atomisation coupled with an internal oxidation step during the HIP,<sup>10</sup> or the in-situ oxidation of an element like titanium in the molten steel with electromagnetically controlled stirring.<sup>11</sup> Other approaches, such as oxide formation in a melt pool,<sup>12</sup> internal oxidation,<sup>13</sup> and spark

\*Corresponding author's e-mail:  
paul.mcguinness@imt.si



© 2024 The Author(s). Except when otherwise noted, articles in this journal are published under the terms and conditions of the Creative Commons Attribution 4.0 International License (CC BY 4.0).

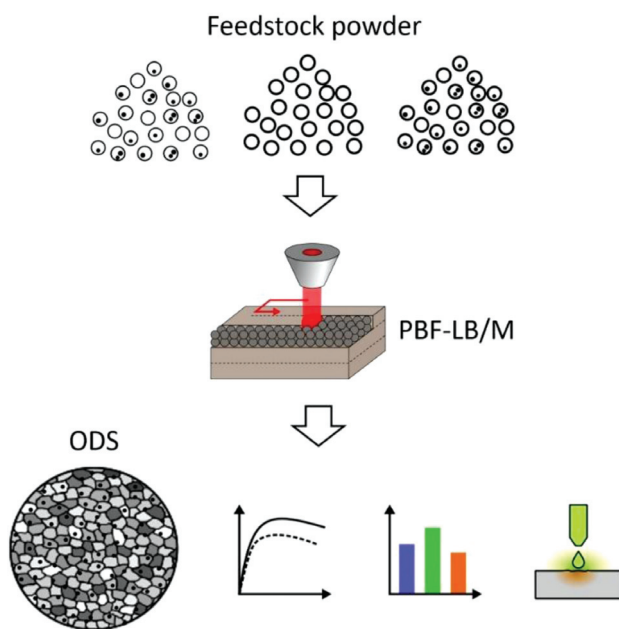
plasma sintering (SPS)<sup>14–16</sup> have also been explored. However, these methods are greatly constrained in terms of the variety of shapes they can produce, often necessitating additional machining or slicing to achieve more complex geometries. Here, we look at how additive manufacturing (AM), in particular selective laser melting (SLM), which is sometimes referred to as laser powder-bed fusion (LPBF) or simply PBF, and more recently directed-energy deposition (DED), have advanced the fabrication of ODS alloys. **Figure 1** schematically illustrates the process of powder preparation as the feedstock material for the PBF printing process, along with a schematic representation of the microstructure with homogeneously distributed nano-oxide particles, which contribute to the high-temperature stability of the material in terms of mechanical, tribological, and corrosion properties.

## 2 EARLY WORK ON THE ADDITIVE MANUFACTURING OF ODS MATERIALS UP TO 2020

Laser-based AM has the very important advantage of being able to realise complex parts directly from the feedstock metal powders.<sup>17</sup> Such a process combines a small melt pool, created by the energy delivered by the laser, with high solidification rates that occur as the laser exits the region of the melt pool, giving them complex flow patterns and a homogenous dispersion of the

sub-micrometer oxide particles.<sup>18</sup> SLM is an AM technique that uses a high-power laser to selectively fuse fine metal powder particles, in a layer-by-layer process, to create a solid, three-dimensional object. For example, as early as 2009, John C. Walker et al. fabricated an ODS PM2000 alloy using SLM, managing to ensure a nano-scale oxide dispersion due to the high cooling rates<sup>19</sup> in a paper that appeared in the journal *Advanced Engineering Materials*. The researchers were attracted by the high solidification cooling rates typically associated with laser welding ( $>10^5$  deg C s<sup>-1</sup>). Their conclusion was that optimization of the SLM processing parameters could lead to the retention of properties in a dispersion-strengthened microstructure, and suggested that the applications of SLM-built PM2000 parts could include custom-made orthopedic hip joints, high-temperature heat exchangers, and chemical tubing, as well as conventional furnace-furniture components. In 2015, Boegelein et al. continued the work on PM2000 and reported on the mechanical response and deformation mechanisms of ferritic ODS steel structures produced by SLM.<sup>20</sup> They found that a post-build anneal resulted in an increase in the yield strength of wall builds up to values observed for conventionally fabricated, recrystallised PM2000. In 2016, the same group characterised a complex, thin-walled structure fabricated by SLM using a ferritic ODS steel.<sup>21</sup> Also in 2015, Ryan M. Hunt et al. produced a MA956 oxide-dispersion-strengthened steel by SLM, achieving 97 % of the wrought MA956's density, but only 65 % of the wrought strength.<sup>22</sup> 2018 saw the publication of a paper on the AM of a 316L steel-matrix composite reinforced with CeO<sub>2</sub> particles, which involved adjusting the laser scanning speed to optimise the process by Salman et al.<sup>23</sup> Their work led to some important conclusions. First, the addition of CeO<sub>2</sub> does not change the phase formation during solidification, although it affects the microstructure of the composite, which is greatly refined compared to the unreinforced AISI 316L. The refined microstructure strengthens the composite without adversely affecting the plastic deformation. These results indicated that dense AISI 316L/CeO<sub>2</sub> composites with enhanced room-temperature strength could be synthesized by SLM.

In 2016, H. Springer et al. described an efficient AM production of oxide- and nitride-dispersion-strengthened materials based on atmospheric reactions in liquid-metal deposition.<sup>24</sup> They presented an alternative production method based on the oxide and nitride formation during liquid-metal-deposition procedures in oxygen- and/or nitrogen-containing atmospheres. Rapid solidification of the small liquid zone suppressed the floatation and agglomeration of particles, while subsequent thermo-mechanical treatments densified the material and helped with particle dispersion. The *in-situ* particle formation, coupled with the high deposition rates, ensured a shortened production chain. The feasibility of the method was demonstrated on austenitic stainless steel and commer-



**Figure 1:** Schematic representation of the production of ODS steel using the PBF-LB/M process. The feedstock powder material is a metallic powder mixed with Y-oxide nanoparticles or other thermally stable oxides. The feedstock powder can also be oxidised, introducing oxide particles into the microstructure during LBF-LB/M. Additionally, a combination of the two methods can be used. The final result is ODS steel with nano-oxide particles uniformly distributed in the microstructure. Such material generally exhibits improved mechanical, tribological, and corrosion properties, particularly at elevated temperatures.

cially available deposition techniques as used in AM, performed without a shielding gas. Even without substantial optimisation of the processes and material, more than 2  $\varphi$ % of hard and stable  $\text{Cr}_2\text{N}$  particles with sizes down to 80 nm could be evenly dispersed, resulting in pronounced strengthening at room temperature and 700 °C without any significant loss in ductility.

In the latter part of 2017, an important paper by C. Kenel et al. on the SLM of an ODS  $\gamma$ -TiAl alloy in an attempt to produce complex structures was published in the journal *Materials & Design*.<sup>25</sup> They found that the SLM material consisted mainly of an  $\alpha_2$  phase with embedded Y-rich dispersoids and that the cracking frequency was reduced for a high radiant exposure. Indeed, re-scanning with 90° reduced the degree of cracking by up to a factor of approximately 2. In 2018, Streubel et al. published a report indicating that laser-generated nanoparticles were deposited on powders for the AM of ODS alloy parts via laser metal deposition in the Japanese Journal of Applied Physics.<sup>26</sup> They presented a new route for the adsorption of pulsed-laser-dispersed nanoparticles onto metal powders in an aqueous solution without the need for any binders or surfactants. Using an electrostatic interaction, they deposited  $\text{Y}_2\text{O}_3$  nanoparticles onto iron–chromium powders and achieved a high dispersion of nano-sized particles in the metallic powders. Using AM they showed how the particle spacing of the oxide inclusions could be adjusted by the initial mass fraction of the adsorbed  $\text{Y}_2\text{O}_3$  particles on the micropowder. The procedure was a robust one for the AM of ODS alloys based on oxide nanoparticles supported on steel micropowders.

A year later, Markus B. Wilms et al. reported on the laser AM of ODS steels using laser-generated nanoparticle-metal composite powders in *Procedia CIRP*.<sup>27</sup> They described a new route for synthesising powder composites suitable for laser AM. The powder composites, consisting of micrometre-sized, stainless-steel powder, homogeneously decorated with nano-scaled  $\text{Y}_2\text{O}_3$  powder particles, were manufactured by laser processing colloids and electrostatic deposition. After consolidation by laser metal deposition and SLM, the resulting specimens showed superior mechanical properties at elevated temperatures caused by the nano-sized, homogeneously distributed dispersoids.

2020 saw C. Doñate-Buendia et al. describe the effect of nanoparticle addition on the microstructure and microhardness of ODS steels produced by laser powder-bed fusion (PBF) and directed-energy deposition (DED).<sup>28</sup> The powder composites were made of micrometre-sized iron-chromium powders that were homogeneously decorated with  $\text{Y}_2\text{O}_3$  nanoparticles, synthesized by pulsed-laser fragmentation in water. When consolidated by PBF and DED, an enhanced microhardness was measured, which was related to the microstructural differences between the differently processed samples.

The appropriateness of DED was demonstrated several times using mechanically alloyed powder material. For example, in 2018, Zheng Min et al., in *The Journal of Engineering for Gas Turbines and Power*, published an article on the fabrication and characterisation of an additive-manufactured, nickel-based, ODS coating layer.<sup>29</sup> They detected the formation of an  $\text{Al}_2\text{O}_3$  scale above the ODS coating layer at an early stage. This had long-term stability throughout the oxidation test. The formation of a stable alumina scale acted as a protective layer to prevent the oxygen from penetrating the top surface. Spallation of part of the nickel and chromium oxides was observed, but the thickness of the oxide scale showed almost no change. In addition, they observed adhesion between the ODS coating layer while the substrate was stable throughout the oxidation test. The study has proved that AM can be used to fabricate structural and protective coating layers for turbine airfoils. A year later, Silja-Katharina Rittinghaus & Markus B. Wilms, described the ODS of  $\gamma$ -TiAl by laser MM in the *Journal of Alloys and Compounds*.<sup>30</sup> They looked at a short-milled powder compound consisting of gas atomized  $\gamma$ -TiAl powder and nano-scaled  $\text{Y}_2\text{O}_3$  powder via DED. They observed fully dense, crack-free samples with homogeneously dispersed  $\text{Y}_2\text{O}_3$  particles. Just prior to this, Barton Mensah Arkhurst et al. published a paper on the DED of 14Cr ODS steel powders using  $\text{Y}_2\text{O}_3$  and  $\text{HfO}_2$  dispersoids,<sup>31</sup> where they detected the presence of nanometer-sized particles in all the deposition layers of both  $\text{Y}_2\text{O}_3$ - and  $\text{HfO}_2$ -dispersed ODS steels, and their number densities were far lower than those in conventional bulk ODS steels. However, transmission electron microscopy analyses revealed that the dispersion and retention of the nanoparticles within the melt were not achieved, even with  $\text{HfO}_2$  as a dispersoid. Furthermore, the deposition layers of both the as-milled  $\text{Y}_2\text{O}_3$  and  $\text{HfO}_2$  ODS steels also exhibited an unusual nano-grained structure. The microhardnesses of the  $\text{HfO}_2$ - and the  $\text{Y}_2\text{O}_3$ -dispersed ODS steels in both the as-milled and the pre-annealed conditions were higher than the substrate.

2018 also saw the publication of an article on the laser AM of ODS steels using laser-generated, nanoparticle, metal-composite powders<sup>28</sup> as part of the 10th CIRP Conference on Photonic Technologies. A new route for the synthesis of powder composites suitable for processing with laser AM was described. The powder composites, consisting of micrometer-sized, stainless-steel powder, homogeneously decorated with nano-scaled  $\text{Y}_2\text{O}_3$  powder particles, were manufactured by the laser processing of colloids and electrostatic deposition. After consolidation by laser metal deposition and SLM, the resulting specimens showed superior mechanical properties at elevated temperatures, caused by the nano-sized, homogeneously distributed dispersoids.

The agglomeration of oxide nanoparticles in DED-manufactured material was found to be problematic. Yingnan Shi et al. conducted a microstructure char-



acterization and measured the mechanical properties of a laser-engineered, net-shaped ODS Fe-9Cr alloy and found that equiaxed martensitic grains together with micro-pores were observed in the as-deposited alloys.<sup>32</sup> However, with an increase in laser power, the density and size of the pores decreased, the grain size of the alloys increased, while the number density of nanoscale oxides decreased due to the formation of thicker, Y-Ti-O-enriched and Al-O-enriched slag layers. Hot isostatic pressing (HIP) processing of the as-deposited ODS alloy with a low laser power reduced the density and the size of the micro-pores, refined the grain size, and precipitated a higher density of nanoscale oxides ( $\text{Y}_2\text{TiO}_5$  and  $\text{Y}_2\text{Ti}_2\text{O}_7$ ). This improved the tensile properties compared to ODS EUROFER steel produced by conventional powder metallurgy. A second paper by Yingnan Shi et al. in early 2020 in the journal *Materials Science and Engineering: A*<sup>33</sup> looked at the microstructure and tensile properties of a Zr-containing ODS FeCrAl alloy fabricated by laser AM. The findings included coarse columnar grains with preferred orientation ([001] fibre texture), obtained after deposition, were retained after a post-build heat treatment. The average width of the columnar grains for both the as-deposited and heat-treated samples was  $\sim 180\text{--}190\text{ }\mu\text{m}$ . A slag-like layer made up of  $\text{Y}_3\text{Al}_5\text{O}_{12}$ ,  $\text{ZrO}_2$  and  $\text{Al}_2\text{O}_3$  was also observed on the surface of deposited sample. The post-build heat treatment produced high-density nanoscale  $\text{Y}_4\text{Zr}_3\text{O}_{12}$  oxides in the matrix. The agglomeration was attributed to reduced cooling rates that are the result of a larger beam diameter and slower speeds used in DED compared to SLM.

### 3 COMMONLY USED OXIDES IN ODS STEEL PRODUCTION

Oxide-dispersion-strengthened (ODS) steels are designed to withstand extreme environments, such as high temperatures, radiation exposure, and mechanical stress. They are strengthened by the uniform distribution of fine oxide particles within the metallic matrix, which enhances the mechanical and thermal stability, while improving the creep resistance and the high-temperature strength. The choice of oxide plays a crucial role in de-

termining the properties and performance of ODS materials, as each oxide imparts unique characteristics to the alloy.

**Table 1** highlights some of the most used oxides in ODS material production, including yttria ( $\text{Y}_2\text{O}_3$ ), alumina ( $\text{Al}_2\text{O}_3$ ), ceria ( $\text{CeO}_2$ ), and titania ( $\text{TiO}_2$ ). These oxides are selected based on their thermal stability, chemical inertness, and ability to form nanoscale dispersoids that inhibit dislocation movement and grain-boundary sliding. In addition to their role in enhancing the mechanical properties, these oxides contribute to oxidation resistance, radiation tolerance, and microstructural stability, making ODS materials suitable for applications in aerospace, nuclear reactors, and high-temperature industrial processes.

### 4 THE ADDITIVE MANUFACTURING OF ODS MATERIALS SINCE 2020

Since 2020 there has been increasing levels of interest in producing ODS materials using AM. Many of the same research groups have continued to be involved. Markus B. Wilms et al. published an article in the journal *Progress in Additive Manufacturing* about manufacturing ODS steels using an advanced DED process of high-speed laser cladding.<sup>47</sup> They described the manufacturing of an iron-based ODS PM2000 composite material with the chemical composition  $\text{Fe}_{20}\text{Cr}_{4.5}\text{Al}_{0.5}\text{Ti} + 0.5\text{Y}_2\text{O}_3$  (in w/%) using an advanced DED process referred to as high-speed laser cladding (HSLC). The characteristic high solidification rates of the HSLC processes successfully dispersed the nano-scale, yttrium-based oxides in the ferritic stainless-steel matrix. The suppression of nanoparticle agglomeration during the melting stage, which is frequently observed in conventional DED processes of ODS materials, was reflected in smaller dispersoid sizes and corresponding higher hardnesses compared to DED counterparts.

Then in 2022, Markus B. Wilms and Silja-Katharina Rittinghaus switched their attention to copper alloys and published an article on the laser AM of ODS copper-chromium-niobium alloys.<sup>48</sup> The alloy was chosen because of the role it plays in thermally stressed compo-

**Table 1:** Overview of each oxide, including its chemical formula, key characteristics, specific roles in ODS materials.

Oxide	Chemical formula	Key characteristics	Role in ODS material	Citation
Yttria	$\text{Y}_2\text{O}_3$	High thermal stability, excellent resistance to coarsening, and strong oxide-matrix bonding.	Improves high-temperature strength, creep resistance, and oxidation resistance.	<sup>34, 35, 36, 37, 38, 39</sup>
Alumina	$\text{Al}_2\text{O}_3$	High hardness, good wear resistance, and thermal stability.	Enhances mechanical properties, especially wear resistance, and provides thermal stability at high temperatures.	<sup>39, 40, 41, 42, 43</sup>
Ceria	$\text{CeO}_2$	Good thermal and chemical stability, and oxygen storage capacity.	Improves oxidation resistance and stabilizes microstructure at high temperatures.	<sup>44, 45, 46</sup>
Titania	$\text{TiO}_2$	High melting point and good stability in the steel matrix.	Enhances wear resistance and high-temperature strength, contributing to microstructural stability.	<sup>39, 41, 46</sup>

nents in modern aerospace propulsion systems. Such an application requires both material strength and stability at high temperatures, which can be achieved with ODS. The addition of nanoscale  $Y_2O_3$  resulted in a complex microstructure with populations of Cr- and Nb-enriched nanoparticles next to Cr-rich yttrium oxide nanoparticles, homogeneously dispersed in the metallic copper matrix. The evolution of both nanoparticle species with the addition of  $Y_2O_3$  led to a remarkable increase of the hardness compared to their unreinforced counterparts.

Work in the US by Jennifer A. Glerum et al. on the effect of oxide dispersoids on precipitation-strengthened Al-1.7Zr (w/%) alloys produced by laser powder-bed fusion was published in the journal *Additive Manufacturing* in August 2022.<sup>49</sup> In this study Binary Al-1.7 w/% Zr (Al-0.5 at % Zr) alloys, with and without 1 w/% (0.7  $\phi$ %)  $Al_2O_3$  nanoparticle additions (80–100 nm), were fabricated by laser powder-bed fusion (L-PBF) from blends of Al, Zr, and  $Al_2O_3$  powders. They found that the elemental Zr, which is ~80 % dissolved in the melt pool, forms primary  $L1_2$ - $Al_3Zr$  precipitates upon solidification and is retained in a supersaturated solid solution in the Al matrix. The primary  $L1_2$ - $Al_3Zr$  phase takes three forms: (i) discrete, micron-sized precipitates that nucleate fine Al matrix grains, (ii) elongated “strings” of submicron precipitates forming in highly enriched Zr regions, and (iii) < 100 nm precipitates within fine Al-grains. The  $Al_2O_3$  nano-dispersoid additions are either lost to slag or entrapped within the Al-grains as oxide dispersoids, resulting in an oxide concentration of 0.46 w/% for the modified ODS alloy.  $Al_2O_3$  nanoscale dispersoids (0.28 w/%) are also observed in the unmodified alloy, and are assumed to originate from (i) native oxides from the surface of the Al powder particles, and/or (ii) reaction with trace amounts of oxygen in the processing environment. The hardness upon aging at 400 °C reaches a peak value of ~750–850 MPa after 1.5 h, consistent with the precipitation of secondary  $L1_2$ - $Al_3Zr$  nanoprecipitates. With over-aging, the hardness slowly decreased to 500 MPa (the as-processed value) after 1500 h at 400 °C, with no difference observed between the Al-Zr and Al-Zr- $Al_2O_3$  alloys. They reported that the strengthening mechanisms combine Hall-Petch strengthening from micron-sized grains, Zr solid-solution strengthening, and secondary  $L1_2$ - $Al_3Zr$  precipitate strengthening, with slow precipitate coarsening and effective grain pinning by  $Al_3Zr$  precipitates and oxide dispersoids located at the grain boundaries. The creep resistance was comparable for both alloys, which showed threshold stresses of ~15 MPa and 6 MPa, at 300 °C and 400 °C, respectively.

In 2023, researchers in France, Lucas Autones et al., assessed ferritic ODS steels obtained by laser AM, which they published in the journal *Manufacturing and Materials Processing*.<sup>50</sup> This study was to assess the potential of laser AM for the elaboration of ferritic/martensitic ODS steels. These materials are usually man-

ufactured by the mechanical alloying of powders followed by hot consolidation in the solid state. Two Fe-14Cr-1W ODS powders were considered. The first powder was obtained by mechanical alloying, and the second was through soft mixing of an atomized Fe-14Cr steel powder with yttria nanoparticles. The results obtained with the laser powder-bed fusion (LPBF) process were compared to a non-ODS powder and to a conventional ODS material obtained by HIP. The microstructural and mechanical characterizations showed that it is possible to obtain nano-oxides in the material, but their density remains low compared to HIP ODS steels, regardless of the initial powders considered. The ODS obtained using laser AM had mechanical properties that were modest compared to conventional ODS.

In 2023, Wilms et al.<sup>51</sup> reported again on the AM of ODS alloys. They looked at the production of suitable powder materials and consolidation techniques including in-situ manufacturing techniques. The main emphasis was process-related influences on the final ODS material and its microstructural features as well as mechanical performance. Different classes of ODS alloys were presented and discussed along with areas of application. They also noted the current drawbacks of ODS alloys, highlighting the development required for the widespread application of these materials. Perhaps the most important conclusion that they draw is that shorter process chain, i.e., combining synthesis and shaping in a single process step, suggests that the economic production of ODS alloys by AM could be feasible.

Late 2023 saw the publication of a new paper by SeungHyeok Chung et al.<sup>52</sup> on the additive manufacturing of ODS CoCrNi medium-entropy alloy by *in-situ* oxide synthesis in the Journal of Alloys and Compounds. They proposed a novel ODS-feedstock preparation strategy using the surface-modification and reinforcement transplant (SMART) process and an *in-situ* dispersoid synthesis strategy that could be applied during DED. The feedstock of the ODS CoCrNi was prepared with a Co-Y alloy powder and NiO or  $TiO_2$  as the Y and O suppliers, respectively. To evaluate the *in-situ* dispersoid synthesis effect, a counterpart was prepared by adding  $Y_2O_3$  using an *ex-situ* dispersoid-addition approach. The addition of  $TiO_2$  to induce the supply of O and Ti led to an *in-situ* synthesis of ODS CoCrNi with a 63.5 % reduction in dispersoid size compared to the pristine CoCrNi resulting in the strengthening of the CoCrNi MEA matrix. This study suggests that ODS alloys with an optimal microstructure can be fabricated using the DED process through *in-situ* dispersoid synthesis aided by Ti and O suppliers.

## 5 CONCLUDING REMARKS

The advancements in additive manufacturing (AM) of oxide-dispersion-strengthened (ODS) alloys have demonstrated the potential for producing materials with

exceptional mechanical properties suitable for high-temperature applications in aerospace, nuclear reactors, and other demanding environments. Selective laser melting (SLM) and directed-energy deposition (DED) are particularly effective AM techniques for fabricating ODS alloys. These methods offer the crucial advantage of producing complex geometries directly from metal powders, thus eliminating the need for extensive post-processing and additional machining required by traditional manufacturing methods. High solidification rates inherent in laser-based AM processes facilitate the homogeneous dispersion of sub-micrometer oxide particles within the metal matrix. This uniform dispersion is essential for the superior mechanical properties of ODS alloys. Optimization of post-build annealing and processing parameters further enhances these properties, leading to improvements in density and microstructural refinement. Research has explored various ODS materials, including Fe-Cr-Al, PM2000, MA956, and 316L steel matrix composites, revealing improvements in density and microstructure. Efforts to produce ODS  $\gamma$ -TiAl alloys have successfully reduced cracking through careful optimization of the manufacturing process. Novel approaches, such as in-situ oxide and nitride formation during the AM process, have simplified the production chain and improved particle dispersion. Techniques like laser-generated nanoparticle deposition onto metal powders and high-speed laser cladding (HSLC) have been particularly successful in enhancing the distribution and retention of oxide particles.

Despite these advancements, the agglomeration of oxide nanoparticles remains a challenge, particularly in DED processes. Continued research is required to refine processing conditions and material compositions to achieve consistent mechanical properties and optimal microstructural features. Recent studies have extended the application of AM techniques to various ODS alloys, including copper-chromium-niobium and Al-Zr alloys. These studies emphasise the importance of combining synthesis and shaping in a single step to make ODS alloy production more economically viable. The potential for economically feasible, large-scale production of ODS alloys using AM techniques is promising, with ongoing efforts to optimise processes and expand the range of applicable materials.

In summary, the progress in the additive manufacturing of ODS alloys highlights advances in material properties and production efficiencies. While challenges remain, the future of ODS alloy fabrication using AM techniques appears bright, with substantial potential for industrial applications.

## Acknowledgment

We would like to acknowledge the project titled "New ODS Steel Structure for Extreme Environments Using the Ultrasonic Dispersion of Nano-Oxides in

Combination with SLM and PPS." This project is funded through the Weave program scheme under the OPUS call, involving collaboration between three countries: Poland, the Czech Republic, and Slovenia, with Poland as the lead country. The project is financed by Poland's National Science Center (NCN) under grant number 2021/43/I/ST8/01018, by the Czech Republic's Grant Agency (GACR) under grant number 22-04227L, and by Slovenia's Agency for Research and Innovation (ARIS) under grant number N2-0276.

## 6 REFERENCES

- <sup>1</sup> R. Xu, Z. Geng, Y. Wu, C. Chen, M. Ni, D. Li, T. Zhang, H. Huang, F. Liu, R. Li, K. Zhou, Microstructure and mechanical properties of in-situ oxide-dispersion-strengthened NiCrFeY alloy produced by laser powder bed fusion, *Advanced Powder Materials*, 1 (2022), 4, 100056, doi:10.1016/j.apmate.2022.100056
- <sup>2</sup> P. Wang, Z. Qi, Q. Li, Y. Zhang, X. Cheng, X. Wu, S. Mei, Simultaneously improving the strength and ductility of an oxide dispersion-strengthened high-entropy alloy by employing innovative precursors for oxide formation, *Journal of Alloys and Compounds*, 1005 (2024), 176192, doi:10.1016/j.jallcom.2024.176192.
- <sup>3</sup> Y. Zhang, K. Lu, F. Jiang, Y. Chen, X. Liang, Superior strength-ductility synergy of an oxide-dispersion strengthened CoCrNi-based multi-principal element alloy. *Materials Research Letters*, 12 (2024) 11, 825–833. doi:10.1080/21663831.2024.2393166
- <sup>4</sup> R. Kocich, L. Kunčická, P. Král, K. Dvořák, Mechanical Behavior of Oxide Dispersion Strengthened Steel Directly Consolidated by Rotary Swaging. *Materials*, 17 (2024) 19, 4831. doi:10.3390/ma17194831
- <sup>5</sup> M. B. Wilms, S.-K. Rittinghaus, M. Goßling, B. Gökce, Additive manufacturing of oxide-dispersion strengthened alloys: Materials, synthesis and manufacturing, *Progress in Materials Science*, 133 (2023), 101049, doi:10.1016/j.pmatsci.2022.101049
- <sup>6</sup> S. M. S. Aghamiri, N. Oono, S. Ukai, R. Kasada, H. Noto, Y. Hishinuma, T. Muroga, Microstructure and mechanical properties of mechanically alloyed ODS copper alloy for fusion material application, *Nuclear Materials and Energy*, 15 (2018), 17–22, doi:10.1016/j.nme.2018.05.019
- <sup>7</sup> P. Unifantowicz, Z. Oksiuta, P. Olier, Y. de Carlan, N. Baluc, Microstructure and mechanical properties of an ODS RAF steel fabricated by hot extrusion or hot isostatic pressing, *Fusion Engineering and Design*, 86 (2011) 9–11, 2413–2416, doi:10.1016/j.fusengdes.2011.01.022.
- <sup>8</sup> Z. Oksiuta, M. Lewandowska, K.J. Kurzydowski, N. Baluc, Influence of hot rolling and high speed hydrostatic extrusion on the microstructure and mechanical properties of an ODS RAF steel, *Journal of Nuclear Materials*, 409 (2011) 2, 86–93, doi:10.1016/j.jnucmat.2010.09.019
- <sup>9</sup> B. Reppich, On the dispersion strengthening mechanisms in ODS materials. *Zeitschrift fuer Metallkunde/Materials Research and Advanced Techniques*. (2002) 93, 605–613
- <sup>10</sup> D. Pazos, A. Cintins, V. de Castro, P. Fernández, J. Hoffmann, W. García Vargas, T. Leguey, J. Purans, A. Anspoks, A. Kuzmin, I. Iturriza, N. Ordás, ODS ferritic steels obtained from gas atomized powders through the STARS processing route: Reactive synthesis as an alternative to mechanical alloying, *Nuclear Materials and Energy*, 17 (2018), 1–8, doi:10.1016/j.nme.2018.06.014
- <sup>11</sup> C. Doñate-Buendía, F. Frömel, M. B. Wilms, R. Streubel, J. Tenkamp, T. Hupfeld, M. Nachev, E. Gökce, A. Weisheit, S. Barcikowski, F. Walther, J. H. Schleifenbaum, B. Gökce, Oxide dispersion-strengthened alloys generated by laser metal deposition of laser-generated nanoparticle-metal powder composites, *Materials & Design*, 154 (2018), 360–369, doi:10.1016/j.matdes.2018.05.044.



- <sup>12</sup> C. Dai, C. Schade, D. Apelian, et al. Processing Techniques for ODS Stainless Steels. *Metall Mater Trans B* 49 (2018), 3043–3055, doi:10.1007/s11663-018-1429-y
- <sup>13</sup> T. Kaito, T. Narita, S. Ukai, Y. Matsuda, High temperature oxidation behavior of ODS steels, *Journal of Nuclear Materials*, 329–333B (2004), 1388–1392, doi:10.1016/j.jnucmat.2004.04.203
- <sup>14</sup> E. Macía, A. García-Junceda, M. Serrano, S. J. Hong, M. Campos, Effect of mechanical alloying on the microstructural evolution of a ferritic ODS steel with (Y–Ti–Al–Zr) addition processed by Spark Plasma Sintering (SPS), *Nuclear Engineering and Technology*, 53 (2021) 8, 2582–2590, doi:10.1016/j.net.2021.02.002
- <sup>15</sup> X. Zhou, Y. Liu, L. Yu, Z. Ma, Q. Guo, Y. Huang, H. Li, Microstructure characteristic and mechanical property of transformable 9Cr-ODS steel fabricated by spark plasma sintering, *Materials & Design*, 132 (2017), 158–169, doi:10.1016/j.matdes.2017.06.063.
- <sup>16</sup> J. Fu, J.C. Brouwer, I. M. Richardson, M. J. M. Hermans, Effect of mechanical alloying and spark plasma sintering on the microstructure and mechanical properties of ODS Eurofer, *Materials & Design*, 177 (2019), 107849, doi:10.1016/j.matdes.2019.107849
- <sup>17</sup> L. E. T. Mathias, V. E. Pinotti, B. F. Batistão, et al. Metal powder as feedstock for laser-based additive manufacturing: From production to powder modification. *Journal of Materials Research* 39 (2024), 19–47, doi:10.1557/s43578-023-01271-8
- <sup>18</sup> X. Zhang, C. J. Yocom, B. Mao, Y. Liao, Microstructure evolution during selective laser melting of metallic materials: A review. *Journal of Laser Applications*. 31 (2019), 31201. 10.2351/1.5085206
- <sup>19</sup> J. Walker, K. Berggreen, A. R. Jones, C. Sutcliffe, Fabrication of Fe–Cr–Al Oxide Dispersion Strengthened PM2000 Alloy Using Selective Laser Melting. *Advanced Engineering Materials*. 11 (2009), 541–546. 10.1002/adem.200800407
- <sup>20</sup> T. Boegelein, S. Dryepondt, A. Pandey, K. Dawson, G. Tatlock, Mechanical response and deformation mechanisms of ferritic oxide dispersion strengthened steel structures produced by selective laser melting. *Acta Materialia*. (2015), 87. 10.1016/j.actamat.2014.12.047
- <sup>21</sup> T. Boegelein, E. Louvis, K. Dawson, G. J. Tatlock, A. R. Jones, Characterisation of a complex thin walled structure fabricated by selective laser melting using a ferritic oxide dispersion strengthened steel, *Materials Characterization*, 112 (2016), 30–40, doi:10.1016/j.matchar.2015.11.021
- <sup>22</sup> R. M. Hunt, K. J. Kramer, B. El-Dasher, Selective laser sintering of MA956 oxide dispersion strengthened steel. *Journal of Nuclear Materials* 464 (2015), 80–85. doi:10.1016/j.jnucmat.2015.04.011
- <sup>23</sup> O. O. Salman, A. Funk, A. Waske, J. Eckert, S. Scudino, Additive Manufacturing of a 316L Steel Matrix Composite Reinforced with CeO<sub>2</sub> Particles: Process Optimization by Adjusting the Laser Scanning Speed. *Technologies* (2018), 6, 25. doi:10.3390/technologies6010025
- <sup>24</sup> H. Springer, C. Baron, A. Szczepaniak, E.A. Jägle, M.B. Wilms, A. Weisheit, D. Raabe, Efficient additive manufacturing production of oxide- and nitride-dispersion-strengthened materials through atmospheric reactions in liquid metal deposition, *Materials & Design*, Volume 111, 2016, 60–69, doi:10.1016/j.matdes.2016.08.084
- <sup>25</sup> C. Kenel, G. Dasargyri, T. Bauer, A. Colella, A. B. Spierings, C. Leinenbach, K. Wegener, Selective laser melting of an oxide dispersion strengthened (ODS)  $\gamma$ -TiAl alloy towards production of complex structures, *Materials & Design*, 134 (2017), 81–90, doi:10.1016/j.matdes.2017.08.034
- <sup>26</sup> R. Streubel, M. Wilms, C. Doñate-Buendía, A. Weisheit, S. Barcikowski, J. Schleifenbaum, B. Gökce, Depositing laser-generated nanoparticles on powders for additive manufacturing of oxide dispersed strengthened alloy parts via laser metal deposition. *Japanese Journal of Applied Physics*. (2018), 57, 040310. doi:10.7567/JJAP.57.040310
- <sup>27</sup> M. Wilms, R. Streubel, F. Stern, A. Weisheit, J. Tenkamp, F. Walther, S. Barcikowski, J. Schleifenbaum, B. Gökce, Laser additive manufacturing of oxide dispersion strengthened steels using laser-generated nanoparticle-metal composite powders, *Procedia CIRP*. 74 (2018), 196–200, 10.1016/j.procir.2018.08.093
- <sup>28</sup> C. Doñate-Buendía, R. Streubel, P. Kürnsteiner, M. Wilms, F. Stern, J. Tenkamp, E. Bruder, S. Barcikowski, B. Gault, K. Durst, J. Schleifenbaum, F. Walther, B. Gökce, Effect of nanoparticle addition on the microstructure and microhardness of oxide dispersion strengthened steels produced by laser powder bed fusion and directed energy deposition. *Procedia CIRP*. (2020). 94. doi:10.1016/j.procir.2020.09.009
- <sup>29</sup> Z. Min, S. Parbat, L. Yang, B. Kang, M. Chyu, Fabrication and Characterization of Additive Manufactured Nickel-Based Oxide Dispersion Strengthened Coating Layer for High-Temperature Application. *Journal of Engineering for Gas Turbines and Power*. (2017). 140. doi:10.1115/1.4038351
- <sup>30</sup> S.-K. Rittinghaus, M. Wilms, Oxide dispersion strengthening of  $\gamma$ -TiAl by laser additive manufacturing. *Journal of Alloys and Compounds*. (2019). 804. 10.1016/j.jallcom.2019.07.024
- <sup>31</sup> B. Arkhurst, J.-J. Park, C.-H. Lee, J. H. Kim. Direct Laser Deposition of 14Cr Oxide Dispersion Strengthened Steel Powders Using Y<sub>2</sub>O<sub>3</sub> and HfO<sub>2</sub> Dispersoids. *Journal of Korean Institute of Metals and Materials*. 55 (2017), 550–558, 10.3365/KJMM.2017.55.8.550
- <sup>32</sup> Y. Shi, Z. Lu, H. Xu, R. Xie, Y. Ren, G. Yang, Microstructure characterization and mechanical properties of laser additive manufactured oxide dispersion strengthened Fe-9Cr alloy, *Journal of Alloys and Compounds*, 791 (2019), 121–133, doi:10.1016/j.jallcom.2019.03.284
- <sup>33</sup> Y. Shi, Z. Lu, L. Yu, R. Xie, Y. Ren, G. Yang, Microstructure and tensile properties of Zr-containing ODS-FeCrAl alloy fabricated by laser additive manufacturing, *Materials Science and Engineering: A*, 774 (2020), 138937, doi:10.1016/j.msea.2020.138937
- <sup>34</sup> Duy Nghia Luu, Wei Zhou, Sharon Mui Ling Nai, Influence of nano-Y<sub>2</sub>O<sub>3</sub> addition on the mechanical properties of selective laser melted Inconel 718, *Materials Science and Engineering: A*, 845 (2022), 143233, doi:10.1016/j.msea.2022.143233.
- <sup>35</sup> D. Sakuma, S. Yamashita, K. Oka, S. Ohnuki, L. E. Rehn, E. Wakai, Y<sub>2</sub>O<sub>3</sub> nano-particle formation in ODS ferritic steels by Y and O dual ion-implantation. *Journal of Nuclear Materials*. 329 (2004), 392–396, 10.1016/j.jnucmat.2004.04.039
- <sup>36</sup> L. Zhang, S. Ukai, T. Hoshino, S. Hayashi, X. Qu, Y<sub>2</sub>O<sub>3</sub> evolution and dispersion refinement in Co-base ODS alloys. *Acta Materialia*. 57 (2009), 3671–3682, 10.1016/j.actamat.2009.04.033
- <sup>37</sup> S. Sivasankaran, E.-S. M. Sherif, H. R. Ammar, A. S. Alaboodi, A.-b.H. Mekky, Influence of Oxide Dispersions (Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>, and Y<sub>2</sub>O<sub>3</sub>) in CrFeCuMnNi High-Entropy Alloy on Microstructural Changes and Corrosion Resistance. *Crystals* (2023), 13, 605, doi:10.3390/cryst13040605
- <sup>38</sup> Y. Sun, Q. Liu, S.Z. Diao, F.Q. Zhao, N.H. Oono, S. Ukai, S. Ohnuki, H.H. Zhu, Y. Wu, F.R. Wan, Q. Zhan, Response of oxide nano-particles to helium ion irradiation in oxide dispersion strengthened steels with CeO<sub>2</sub> and Y<sub>2</sub>O<sub>3</sub> additions, *Journal of Nuclear Materials*, 605 (2025), 155580, doi:10.1016/j.jnucmat.2024.155580.
- <sup>39</sup> E. Simondon, P.-F. Giroux, J. Ribis, G. Spartacus, L. Chaffron, T. Gloriant, Innovative method of ODS steels manufacturing by direct introduction of pyrochlore phase through milling, *Materials Characterization*, 181 (2021), 111461, doi:10.1016/j.matchar.2021.111461
- <sup>40</sup> O. Khalaj, H. Jirková, B. Mašek, J. Svoboda. Microstructure Evaluation of New ODS Alloys with Fe-Al Matrix and Al<sub>2</sub>O<sub>3</sub> Particles. In *Proceedings of the 2017 International Conference on Industrial Design Engineering (ICIDE 2017)*. Association for Computing Machinery, New York, NY, USA, (2017), 11–15, doi:10.1145/3178264.3178273
- <sup>41</sup> T. Huang, Y. Shen, Al<sub>2</sub>O<sub>3</sub> Regions/Grains in ODS Steel PM2000 Irradiated With Fe Ions at 700 °C. *Metall Mater Trans A* 54 (2023), 952–961, doi:10.1007/s11661-022-06947-0
- <sup>42</sup> Q. Zhao, L. Yu, Y. Liu, Y. Huang, Q. Guo, H. Li, J. Wu, Evolution of Al-containing phases in ODS steel by hot pressing and annealing. *Powder Technology*. (2017), 311. 10.1016/j.powtec.2017.02.016
- <sup>43</sup> O. Khalaj, H. Jirková, B. Mašek, J. Svoboda, Microstructure Evaluation of New ODS Alloys with Fe-Al Matrix and Al<sub>2</sub>O<sub>3</sub> Particles. (2017), 11–15, 10.1145/3178264.3178273

- <sup>44</sup> R. Husák, H. Hadraba, Z. Chlup, M. Heczko, T. Kruml, V. Puchý, ODS EUROFER Steel Strengthened by Y-(Ce, Hf, La, Sc, and Zr) Complex Oxides. *Metals*, **(2019)** 9, 1148, doi:10.3390/met9111148
- <sup>45</sup> J. Ribis, 1.09 – Phase Stability in Irradiated Alloys, Editor(s): J. M. Rudy Konings, R. E. Stoller, *Comprehensive Nuclear Materials* (Second Edition), Elsevier, 2020, 265–309, doi:10.1016/B978-0-12-803581-8.11647-9
- <sup>46</sup> Y. Uchida, S. Ohnuki, N. Hashimoto, T. Suda, T. Nagai, T. Shibayama, K. Hamada, N. Akasaka, S. Yamashita, S. Ohstuka, T. Yoshitake, Effect of Minor Alloying Element on Dispersing Nanoparticles in ODS Steel. *MRS Proceedings*. **(2011)**. 981. doi:10.1557/PROC-981-0981-JJ07-09
- <sup>47</sup> M. B., Wilms, N. Pirch, B. Gökce, Manufacturing oxide-dispersion-strengthened steels using the advanced directed energy deposition process of high-speed laser cladding. *Prog Addit Manuf* 8, **(2023)**, 159–167, doi:10.1007/s40964-022-00319-1
- <sup>48</sup> M. Wilms, S.-K. Rittinghaus, Laser Additive Manufacturing of Oxide Dispersion-Strengthened Copper-Chromium-Niobium Alloys. *Journal of Manufacturing and Materials Processing*, **(2022)**, 6, doi:10.3390/jmmp6050102
- <sup>49</sup> J. A. Glerum, A. De Luca, M. L. Schuster, C. Kenel, C. Leinenbach, D. C. Dunand, Effect of oxide dispersoids on precipitation-strengthened Al-1.7Zr (wt %) alloys produced by laser powder-bed fusion, *Additive Manufacturing*, 56 **(2022)**, 102933, doi:10.1016/j.addma.2022.102933
- <sup>50</sup> L. Autones, P. Aubry, J. Ribis, H. Leguy, A. Legris, Y. de Carlan, Assessment of Ferritic ODS Steels Obtained by Laser Additive Manufacturing. *Materials* **(2023)** 16, 2397. doi:10.3390/ma16062397
- <sup>51</sup> M. B. Wilms, S.-K. Rittinghaus, M. Goßling, B. Gökce, Additive manufacturing of oxide-dispersion strengthened alloys: Materials, synthesis and manufacturing, *Progress in Materials Science*, 133 **(2023)**, 101049, ISSN 0079-6425, doi:10.1016/j.pmatsci.2022.101049
- <sup>52</sup> S. H. Chung, T. Lee, W. Jeong, B. Seo Kong, H. Jin Ryu, Additive manufacturing of oxide dispersion-strengthened CoCrNi medium-entropy alloy by in situ oxide synthesis, *Journal of Alloys and Compounds*, 965 **(2023)**, 171340, doi:10.1016/j.jallcom.2023.171340