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ORIGINAL REPORT



Superior strength-ductility synergy of an oxide-dispersion strengthened CoCrNi-based multi-principal element alloy

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ABSTRACT

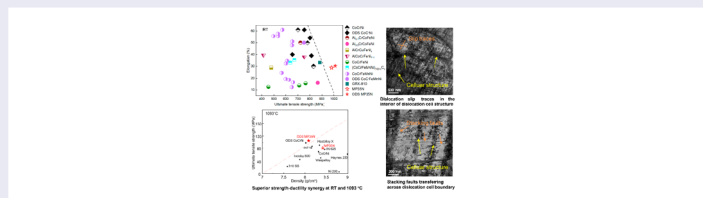
CoCrNi-based multi-principal element alloys (MPEAs) often lack strength at elevated temperatures. Here, to overcome this deficiency, a novel oxide-dispersion strengthening (ODS) CoCrNi-based MPEA was designed by incorporating nano-scale yttria via LPBF. The ODS MPEA possesses superior strength-ductility synergy at both ambient and elevated temperatures to most reported CoCrNi-based MPEAs and state-of-the-art superalloys. TEM investigations revealed dislocation forest and slip traces in the interior of solidification dislocation cell structure, along with extended stacking faults and nano-twins. These features contributed to the excellent strength-ductility of the MPEA. Lastly, quantitative analyses uncovered the contributions of operating strengthening mechanisms to its high strength.

ARTICLE HISTORY

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KEYWORDS

Multi-principal element alloy (MPEA); Oxide-dispersion strengthening (ODS); Transmission electron microscopy (TEM); Dislocation; Laser powder bed fusion (LPBF)



1. Introduction

During the last two decades, multi-principal element alloys (MPEAs), consisting of three to five principal elements, have been gaining increasing interest. Among the huge families of MPEAs, the most investigated face-centered cubic (FCC) single-phase MPEAs are the CoCrNi-based alloys (such as CoCrNi, and CoCr-FeMnNi) [1–5]. The CoCrNi-based alloys are regarded as promising candidates for next-generation engineering applications due to their excellent combination of mechanical properties [2], fatigue [6–8] and fracture resistance [9] at ambient and cryogenic temperatures. The excellent mechanical properties of the alloys are attributed to the relatively low stacking fault energies (SFEs) and the high elastic parameters [2]. However, due to the single-phase microstructure, the limited strength

of CoCrNi-based FCC MPEAs limits their wide applications at elevated temperatures [10–12].

To overcome this limitation, several investigations have added Al and Ti elements into CoCrNi-based MPEAs to enhance high temperature strength (through forming secondary gamma prime phase) and essentially created multi-principal element superalloys [13, 14]. However, the addition of these light-weight elements alters the MPEA's matrix and thus may not take advantage of its unique attributes [15, 16]. Recently, by using additive manufacturing (AM) process that provided an effective microstructural design path to overcoming the strength-ductility tradeoff through introducing hierarchical structures, the oxide dispersion strengthening (ODS) has been introduced in CoCrNi-based MPEAs to improve their strength [15–18]. Through this process,

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the strength and creep properties were increased to a level comparable or even superior to Ni-based superalloys. For instance, Smith et al. [16] fabricated ODS CoCrNi MPEA with significantly increased strength by introducing nano-scale yttria (Y_2O_3) into the CoCrNi matrix using laser powder bed fusion (LPBF). Sahragard-Monfared et al. [15] further found the ODS CoCrNi alloy exhibits superior creep resistance at elevated temperatures due to the interaction of mixed character dislocations with nano-sized oxides.

Recently, by further alloying with heavy refractory elements (such as W), Smith et al. [17] developed an ODS CoCrNi-based MPEA (called ODS GRX-810 alloy) with a twofold improvement in strength and over 1000-fold better creep performance at 1093 °C compared with Ni-based superalloys (such as In718). By further alloying with refractory Mo and W, the current authors [19] designed an FCC MPEA with ultra-high strength (~ 1 GPa) and good ductility ($\sim 30\%$). Therefore, it is of great interest to explore the potential in mechanical properties of CoCrNi-based MPEAs by utilizing the synergistic strengthening effects of nano-oxides [17] and refractory elements [19, 20] (such as Mo and W).

Consequently, in this study, a model CoCrNi-based MPEA, i.e. $\text{Co}_{35.4}\text{Ni}_{35.5}\text{Cr}_{22.9}\text{Mo}_{6.2}$ in at.% and also called MP35N alloy, was used as a matrix alloy to design novel ODS MPEA. It is worth mentioning that the MP35N alloy was selected because of its superior mechanical properties at a wide range of temperatures, and hence was regarded as an excellent candidate for fasteners, springs and plates in aerospace and marine equipment [21]. Finally, the ODS MP35N MPEA was successfully manufactured by LPBF and was found to showcase superior mechanical properties as compared to other reported CoCrNi-based MPEAs and superalloys at ambient and elevated temperatures. The underlying reasons for the superior strength-ductility synergy were also revealed.

2. Experimental procedures

MP35N and ODS MP35N blocks of size of $20 \times 20 \times 10$ mm³ were fabricated by the LPBF process using pre-alloyed MP35N powders and acoustically-mixed powders (consisting of MP35N and 1wt.% Y_2O_3). The detailed compositions of the MP35N powders are listed in Table S1. The average sizes (D_{50}) of the MP35N and Y_2O_3 powder particles were $\sim 45 \mu\text{m}$ and 50 nm, respectively. The morphology and elemental distribution of the mixed powders are shown in Fig. S1. For the LPBF process, an FS271 machine equipped with a Nd: YAG fiber laser and protective Ar gas environment was used. The utilized laser energy density for printing the blocks were

within the range of 80–100 J mm⁻³. Besides, the scan strategy of 67° scan rotation was used for both alloys to reduce the microstructural anisotropy between each layer.

Dog-bone shaped tensile specimens (with gauge length, width and thickness of 8 mm, 2 mm and 1 mm, respectively) were machined from the as-printed blocks by wire electrical discharge machining with testing orientation perpendicular to building direction. Uniaxial tensile tests were performed under strain rate of $5 \times 10^{-4} \text{ s}^{-1}$ and $1.85 \times 10^{-4} \text{ s}^{-1}$ at room temperature (RT) and 1093 °C, respectively. At least two specimens were repeated at each condition to ensure data reliability. Prior to testing, the specimens were ground with SiC paper to remove oxide layers formed during the machining process.

To understand operating deformation mechanisms, the microstructures of the specimens tested before and after the tensile tests were characterized by a Zeiss Sigma 300 scanning electron microscope (SEM, equipped with electron backscattered diffraction (EBSD) detector) and an FEI Talos F200X transmission electron microscope (TEM). Samples for SEM and TEM investigations were prepared by conventional mechanical grinding and subsequent vibratory-polishing. TEM samples were further prepared using a Twin-Jet electro-polisher with an electrolyte of 95% methanol and 5% perchloric acid solution. EBSD imaging was performed with a step size of 1.5 μm , and the data were analyzed using AZtecCrystal software. Conventional bright-field (BF) and dark-field (DF) TEM imaging were performed at 200 kV.

3. Results and discussion

3.1. As-printed microstructure

The as-printed microstructures of the ODS MP35N MPEA obtained from EBSD scanning were shown in Figure 1(a) and 1(b), with the build direction denoted as the Z axis. For comparison, the EBSD results of the MP35N matrix alloy were provided in Fig. S2. Both MP35N and ODS MP35N alloys exhibit equiaxed grains in the XY plane and columnar grains in the XZ plane, along with insignificant texture (Figure 1(a) and 1(b), and Fig. S2a–b). Additionally, the two alloys exhibit similar average grain sizes. Specifically, the average grain sizes are $\sim 28.5 \pm 5 \mu\text{m}$ in the XY plane and the average aspect ratio of the grains in XZ plane is $\sim 3.8 \pm 1.7$ (see Figure 1(a) and 1(b) and Fig. S2(a)–(b)). The similar grain sizes of the two materials allow a relatively-fair comparison of their mechanical properties. Further TEM investigations revealed typical solidification cellular structure formation in the materials (e.g. see Figure 1(c)–(d)). The

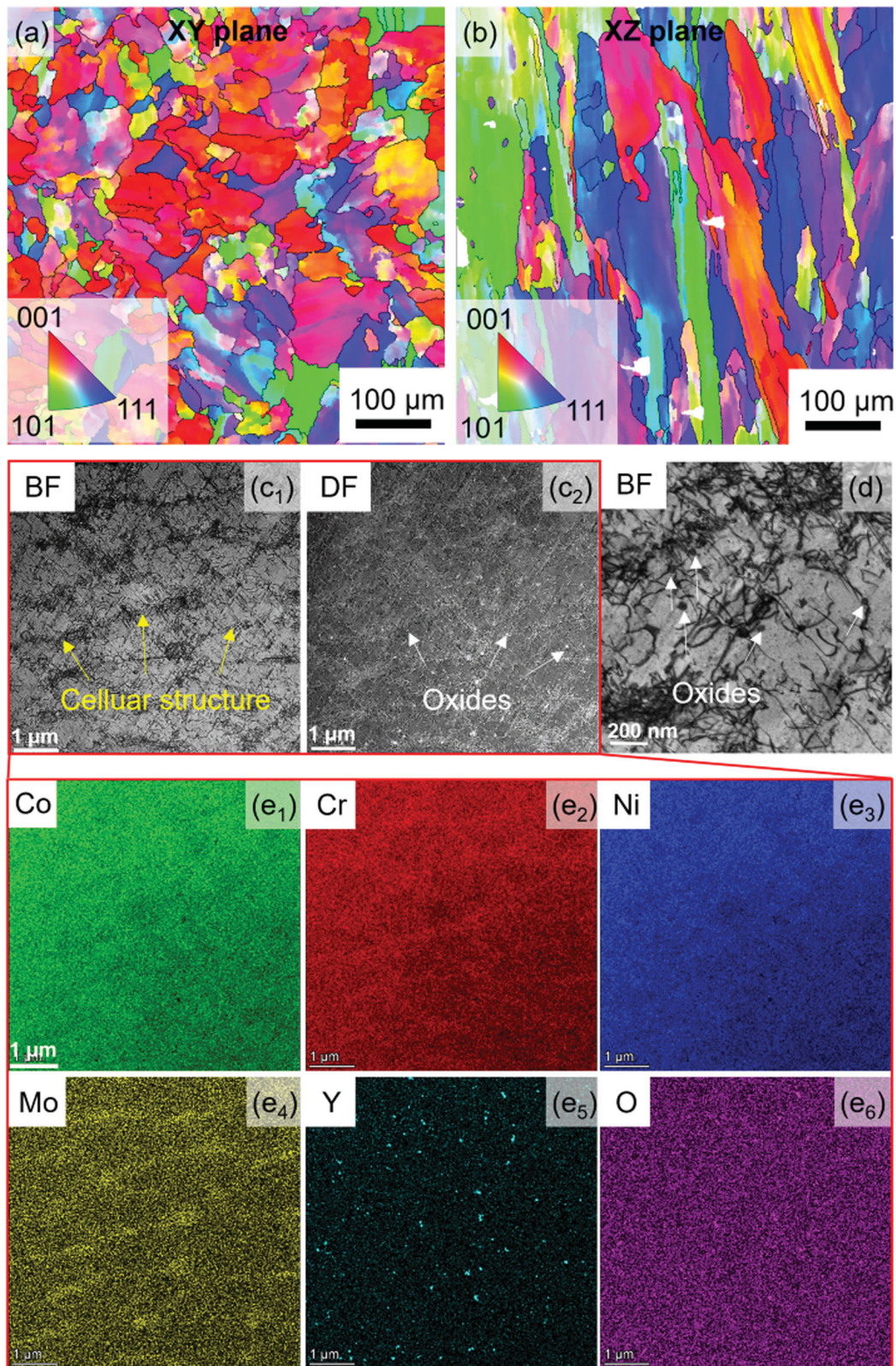


Figure 1. Microstructures of the as-built ODS MP35N MPEA: (a-b) IPF maps along the build direction (z axis) acquired from the XY and XZ planes, (c₁, d) BF-TEM micrographs. (c₂) DF-TEM micrograph and (e₁–e₆) EDS maps were captured at the same region as (c₁).

average size of the cellular structure in the ODS MP35N MPEA is $\sim 1.5 \pm 0.6 \mu\text{m}$. EDS mapping indicated the segregation of Cr and Mo contents into dislocation cell boundaries (see Figure 1e₂), (e₄)).

3.2. Mechanical properties and comparison to other MPEAs

The engineering tensile stress-strain curves and corresponding strain hardening rate curves (SHR) of the investigated materials tested at RT were plotted in Figure 2(a) and (b), respectively. The ODS MP35N MPEA exhibits superior yield strength and ultimate tensile strength (UTS), of $\sim 803 \text{ MPa}$ and 1013 MPa , respectively, see Table S2. These values are higher than its baseline MP35N counterpart (i.e. of 711 MPa and 986 MPa , see Figure 2(a) and Table S2). Herein it is of interest to mention that the as-printed MP35N also shows higher yield strength and UTS than its wrought counterpart (i.e. of $\sim 394 \text{ MPa}$ and 912 MPa , respectively [21]).

Furthermore, the ODS MP35N MPEA also retains good uniform elongation ($\sim 28\%$) and strain hardening ability, which are inherent in the baseline MP35N alloy. Figure 2(b) shows that, the SHR curves of the MP35N and ODS MP35N MPEA exhibit two-stage hardening behavior. Specifically, the SHR shows a rapid drop (at early stage I until $\sim 5\%$), followed by slow decrease (stage II till instable necking) (Figure 2(b)). This behavior is similar to those observed in CoCrNi-based MPEA [2, 10].

Figure 2(c) compares mechanical properties of the two materials tested at 1093°C . The ODS MP35N MPEA exhibits $\sim 28\%$ higher UTS (of $\sim 105 \text{ MPa}$) than the MP35N alloy (of $\sim 82 \text{ MPa}$), along with a satisfactory total elongation ($\sim 12\%$).

To understand how the ODS MP35N MPEA performs as compared to other CoCrNi-based MPEAs and Ni-based superalloys, their mechanical properties tested at RT and 1093°C were plotted in Figure 3(a)–(c), respectively. It can be found that, at RT (Figure 3(a) and 3(b)), the current AM-ed ODS MP35N MPEA exhibits superior strength-ductility synergy as compared to most previously reported CoCrNi-based MPEAs [16–18, 22–30], including as-printed ODS GRX-810 [17], ODS CoCrNi [30] MPEAs and Al-alloyed CoCrFeNi alloys [28, 29]. Moreover, upon comparing the UTS at 1093°C (Figure 3(c)), the ODS MP35N (of $\sim 105 \text{ MPa}$) also outperforms ODS CoCrNi alloy and state-of-the-art superalloys (including IN718 and Haynes 230 [16]).

3.3. Deformation mechanisms

To probe potential reasons for the observed superior strength-ductility synergy, the post-fractured microstructures of ODS MP35N MPEA at RT were characterized

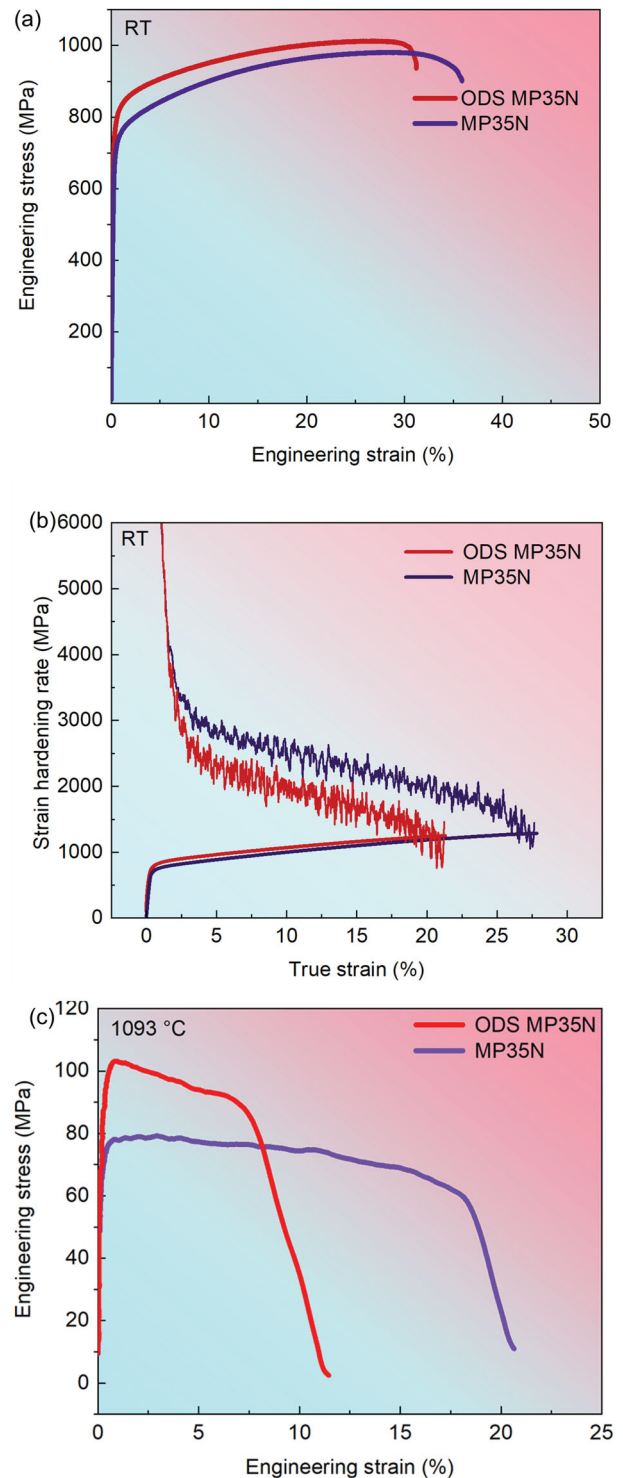


Figure 2. Representative (a) tensile engineering stress-strain curves, (b) strain hardening rate versus true strain curves at RT, and (c) stress-strain curves at 1093°C , for as-built MP35N and ODS MP35N alloys.

tures of ODS MP35N MPEA at RT were characterized via EBSD and TEM. As compared to the as-printed state (Fig. S3), EBSD results in Fig. S4 demonstrated higher KAM values and dislocation motion induced lattice

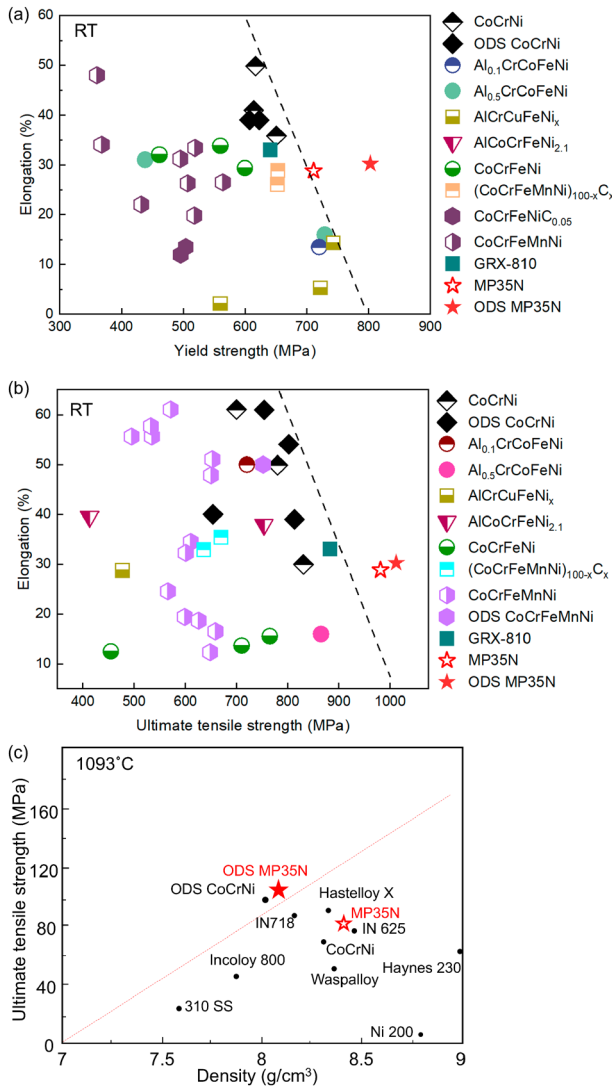


Figure 3. Comparisons of mechanical properties of the current MP35N alloy and ODS MP35N MPEA to other reported as-printed/wrought MPEAs and superalloys at (a-b) RT [16–18, 22–30] and (c) 1093 °C [16].

misorientation after deformation, as indicated by more frequently observed green-colored grains (Fig. S4(b)).

Further in-depth TEM investigations confirmed extensive dislocations activity in the MPEA, as evidenced by a higher dislocation density (compares Figure 4(a) and 4(b) and Figure 1(c)). Moreover, dislocation slip traces and dislocation forest were observed in the interior of dislocation cellular structure (Figure 4(a) and 4(b)), implying dislocation Taylor hardening mechanism. Besides, dislocations were also found to be pinned by the oxides (Figure 4(c)), suggesting operating Orowan hardening mechanism. Apart from the dislocation features, extensive extended stacking faults (SFs, Figure 4(d) and 4(e)) and deformation twins (DTs, Figure 4(f)) were also detected. It appears that the SFs and nano-twins could transfer across the dislocation cell boundaries (Figure 4(d)–(f)).

In light of the observed deformation microstructures, the reasons for the two-stage strain hardening behavior of as-printed ODS MP35N MPEA (Figure 2(b)) can be explained as follows. Deformation initially occurred in the relatively ‘soft’ region (e.g. from dislocation cell boundaries to the interior). It is well-established that dislocations from multiple slip systems are activated to fulfill the von-Mises Criterion and allow compatible deformation with its neighboring grains [31]. Hence, dislocations from different slip systems formed dislocation forest (Figure 4(a) and (b)), contributing to Taylor hardening. Upon further straining, as the number of deformed grains gradually increases, the number of ‘soft’ regions in grains decreases, and hence the strain hardening capability decreased at stage I (Figure 2(b)).

With further straining, most grains exhibit high density of dislocation forest. As applied stress further increases, extended SFs and DTs were activated (Figure 4(d)–(f)). These SFs and DTs contributed to an increased strain hardening by hindering dislocations’ further movement, resulting in enhanced dynamic Hall-Petch effect. Therefore, this leads to a high SHR level and a slow decrease of SHR at stage II (Figure 2(b)). Notably, the above-mentioned deformation behavior, though observed from the ODS MP35N MPEA, is anticipated to be also valid for the MP35N alloy due to their similar strain hardening behavior (Figure 2(b)).

It is of interest to mention that the observed extended SFs and DTs in ODS MP35N were absent in other reported AM-ed CoCrNi-based MPEAs (including CoCrNi [32], ODS CoCrNi [15] and Al_{0.5}CoCrFeNi [28]). The presence of the SFs and DTs in ODS MP35N is attributed to the lower SFE (of ~ 9 mJ/m² [21]) of the matrix as compared to CoCrNi (i.e. 22 ± 4 mJ m⁻² [2]) and Al_{0.5}CoCrFeNi (~ 49 mJ/m² [33]). The lower SFE promotes dislocations dissociation into partial dislocations, which favors the formation of extended SFs and nano twins in the ODS MP35N MPEA. These SFs and DTs contributed to the MPEA’s higher strength-ductility synergy as compared to other CoCrNi-based MPEAs (Figure 3(a)–(c)).

3.4. Strengthening mechanisms

To further quantify the origins of the high strength, the yield strength σ_y of the ODS MP35N MPEA was estimated from the sum of operating strengthening mechanisms, including lattice friction stress σ_{fr} , grain boundary strengthening $\Delta\sigma_g$, dislocation strengthening $\Delta\sigma_d$ and Orowan strengthening $\Delta\sigma_{OR}$, as following equation.

$$\sigma_y = \sigma_{fr} + \Delta\sigma_g + \Delta\sigma_d + \Delta\sigma_{OR} \text{ eq. (1)}$$

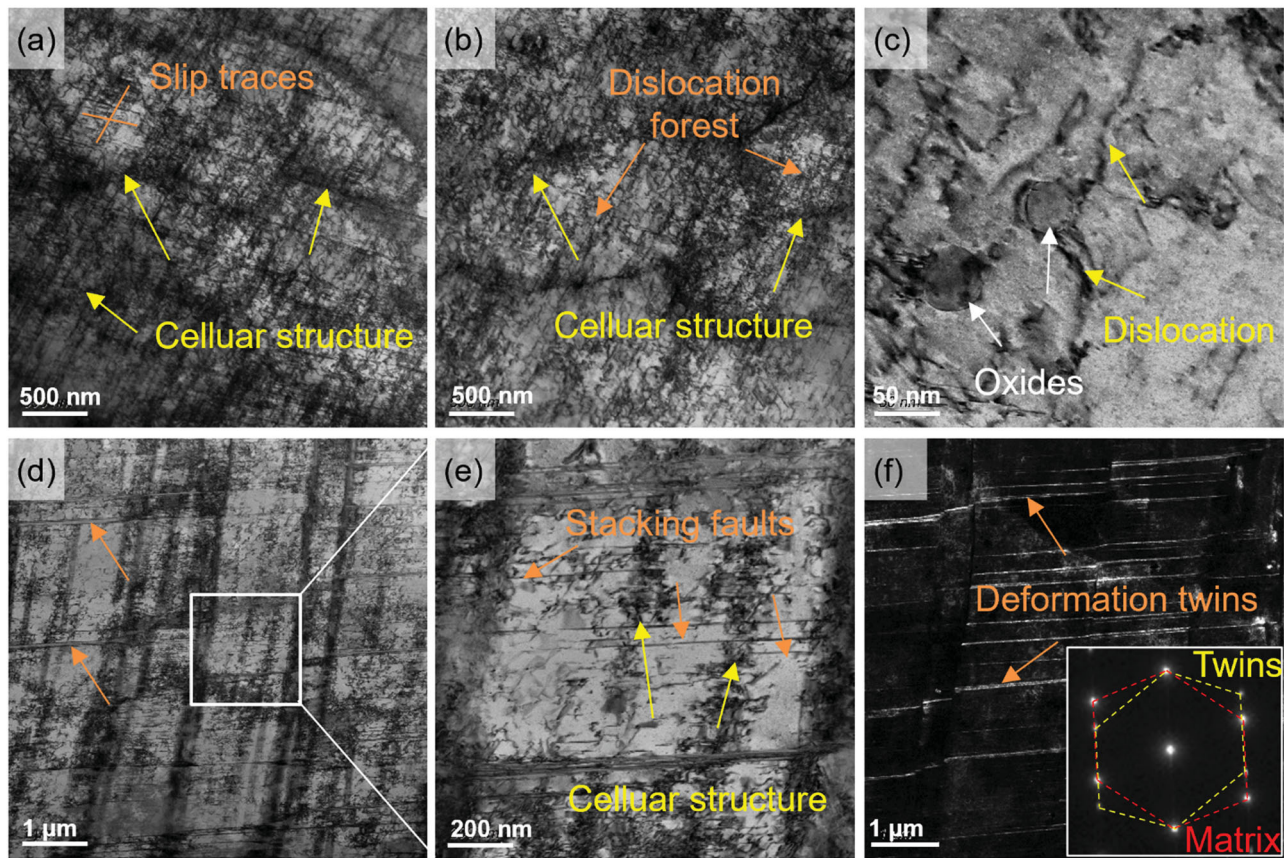


Figure 4. Representative TEM micrographs of the post-fractured ODS MP35N MPEA tested at RT: (a-e) BF-TEM and (f) DF-TEM micrographs. The inset of (f) is a SADP, indicating the presence of nano-twins.

Here σ_{fr} is friction stress from the lattice and solute to the movement of dislocations.

$\Delta\sigma_g$ is expressed as $K_y \cdot d^{-0.5}$, where K_y is Hall-Petch constant signifying the strength of grain boundaries as barriers to dislocation movement, d is average grain size.

$\Delta\sigma_d$ is calculated by $\alpha M G b \sqrt{\rho}$, where α (of ~ 0.4) is an empirical correction factor for FCC materials, M (~ 3.06) is Taylor factor, G is shear modulus, b is Burgers vector of a perfect dislocation, and ρ is dislocation density. The dislocation density can be roughly estimated from the width of dislocation cell boundaries t using $\sqrt{\rho} = 1/t$ [34].

$\Delta\sigma_{OR}$ is estimated from $\frac{0.4M}{\pi\sqrt{1-v}} \times \frac{Gb}{L} \times \ln \frac{\sqrt{\frac{2}{3}} \times d_p}{b}$, where v is Poisson's ratio, d_p is the average size of oxides, L is the average distance between oxides and was estimated from $\sqrt{\frac{2}{3}} \times d_p \left(\sqrt{\frac{\pi}{4v_p}} - 1 \right)$, where v_p is the volume fraction of oxides [35].

The used parameters and constants were given in Table S3. The estimated strength contributors (including σ_{fr} , $\Delta\sigma_g$, $\Delta\sigma_d$, and $\Delta\sigma_{OR}$) along with the estimated and measured yield strength were plotted in Figure 5. Based on the calculation, the theoretically predicted σ_y value

is ~ 788 MPa, which is reasonably consistent with the experimentally measured value (i.e. ~ 803 MPa). Furthermore, it can be seen that the high yield strength of the ODS MP35N originates from the concurrent contributions of lattice friction, grain boundary strengthening, dislocation hardening, and Orowan strengthening. Therefore, the strength of the alloy is higher as compared to other reported MPEAs at RT (such as ODS CoCrNi [16, 30], GRX-810 alloy [17] and Al-alloyed CoCrFeNi alloys [28, 29]). It is worth mentioning that the strength of the ODS CoCrNi-based MPEAs can be further improved by incorporating grain boundary strengthener, such as carbides.

Firstly, the high friction stress of the ODS MP35N MPEA stems from the higher solid solution strengthening of the MP35N matrix alloy, due to the presence of 6.2 at.% large-sized refractory Mo contents, which results in larger lattice distortion (i.e. mean square atomic displacement) than CoCrNi-based MPEAs (including $Al_{0.5}CoCrFeNi$) [19]. Secondly, the high grain boundary strengthening of the ODS MP35N MPEA originates from the relatively low SFE of the matrix alloy (i.e. of ~ 9 mJ/m² [21]) than CoCrNi and $Al_{0.5}CoCrFeNi$ as mentioned before. The low SFE promotes perfect

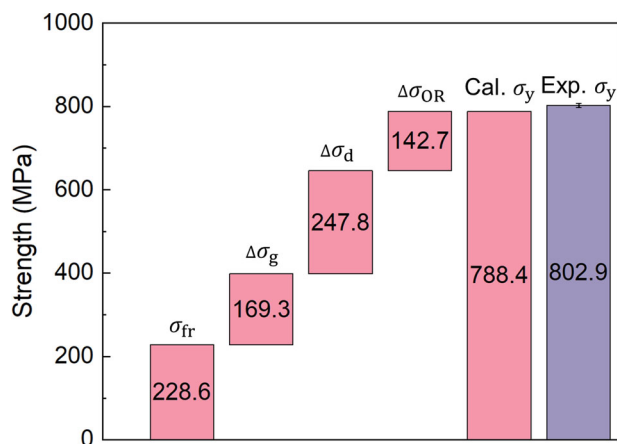


Figure 5. Contributions of operating strengthening mechanism in the ODS MP35N MPEA tested at RT, showing that the calculated σ_y (Cal. σ_y) agrees well with the measured σ_y (Exp. σ_y).

dislocations dissociation (i.e. suppresses partial dislocations recombination) and hence reduces the extent of dislocation cross slip. Consequently, the ability of dislocations transferring across grain boundary via cross slip is reduced [36], which gives rise to a high extent of dislocation pile-up upon grain boundaries and leads to high grain boundary strengthening ability of the investigated ODS MP35N MPEA. Thirdly, the high dislocation strengthening comes from the solidification dislocation cell structure, which is inherent to alloys fabricated by LPBF due to the rapid heating and cooling cycles [32]. The dislocation cell structure can act as dislocation sources to initiate new dislocations and also as barriers to impede their further movement, hence contributing to dislocation hardening. It should be mentioned that the ductility was not significantly affected by the cell structure, as the partial dislocations (along with SFs and DTs) could transfer across the cell boundaries under large applied stresses (Figure 4(d) and 4(e)). Lastly, as the dislocations movement was hindered by the oxides (Figure 4(c)), the Orowan strengthening also played an important role in enhancing the yield strength of ODS MP35N MPEA.

4. Summary

A novel ODS CoCrNi-based (MP35N) MPEA was successfully fabricated via LPBF. The as-built ODS MP35N MPEA exhibits superior strength-ductility synergy at RT and 1093 °C as compared to the as-printed and wrought MP35N matrix alloy, as well as most previously reported CoCrNi-based MPEAs (including ODS CoCrNi, Al_{0.1}CoCrFeNi and Al_{0.5}CoCrFeNi alloys) and advanced Ni-based superalloys (e.g. IN718 and Haynes

230). Microstructural investigations revealed hierarchical structure in the as-built ODS MP35N MPEA, including columnar grains and solidification dislocation cell structure. Post-deformed microstructure of the MPEA exhibits dislocation forest and slip traces in the interior of dislocation cell structure, along with extended stacking faults and nano-twins (that transferred across the cell boundaries). These deformation features together contributed to the good ductility of the ODS MPEA. Further quantitative analyses indicated that the high strength of the alloy originates from the concurrent effects of high lattice friction stress (due to presence of Mo content), grain boundary strengthening (owing to low stacking fault energy as a result of Co content), dislocation strengthening (due to the presence of dislocation cell structure) and oxides Orowan strengthening.

Consequently, this work can serve as a reference for guiding the design of more complex ODS MPEAs with enhanced strength-ductility synergy and also for understanding deformation mechanisms of ODS MPEAs fabricated by LPBF.

CRedit authorship contribution statement

Yiqing Zhang: Methodology, Investigation, Writing-review & editing.

Kaiju Lu: Conceptualization, Formal analysis, Methodology, Investigation, Funding acquisition, Supervision, Writing-original draft.

Fengchun Jiang: Resources, Supervision, Writing-review & editing.

Yongxiong Chen: Methodology, Writing-review & editing.

Xiubing Liang: Resources, Methodology, Supervision, Writing-review & editing.

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Disclosure of interest

The authors report there are no competing interests to declare.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author [K.L.] upon reasonable request.

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