Discovery of a novel low-cost medium-entropy stainless steel with exceptional mechanical behavior over a wide temperature range

Tong Shen (tdshen@ysu.edu.cn)  
Yanshan University  https://orcid.org/0000-0002-0391-3361

Kangkang Wen  
https://orcid.org/0009-0006-9687-4483

Xuecheng Cai  
The University of Hong Kong

B. Sun  
Yanshan University

Rui Feng  
University of Tennessee at Knoxville

Congcong Du  
Yanshan University

Huihui Zhu  
University of Science and Technology Beijing

Ke An  
Oak Ridge National Laboratory  https://orcid.org/0000-0002-6093-429X

Yuan Wu  
University of Science and Technology Beijing  https://orcid.org/0000-0001-7857-0247

Shuaijun Ding  
Yanshan University

Fanxi Meng  
Yanshan University

S. Xin  
Yanshan University

Zhenhua Bai  
Yanshan University

Peter Liaw  
University of Tennessee at Knoxville  https://orcid.org/0000-0003-0185-3411

Physical Sciences - Article
This manuscript has been authored by UT-Battelle, LLC under Contract No. DE-AC05-00OR22725 with the U.S. Department of Energy. The United States Government retains and the publisher, by accepting the article for publication, acknowledges that the United States Government retains a non-exclusive, paid-up, irrevocable, worldwide license to publish or reproduce the published form of this manuscript, or allow others to do so, for United States Government purposes. The Department of Energy will provide public access to these results of federally sponsored research in accordance with the DOE Public Access Plan (http://energy.gov/downloads/doe-public-access-plan).
Discovery of a novel low-cost medium-entropy stainless steel with exceptional mechanical behavior over a wide temperature range

Kangkang Wen\textsuperscript{1,7}, Xuecheng Cai\textsuperscript{2,7}, Baoru Sun\textsuperscript{1,*}, Rui Feng\textsuperscript{3}, Congcong Du\textsuperscript{4}, Huihui Zhu\textsuperscript{5}, Ke An\textsuperscript{3}, Yuan Wu\textsuperscript{3}, Shuaijun Ding\textsuperscript{1}, Fanxi Meng\textsuperscript{1}, Shengwei Xin\textsuperscript{1}, Zhenhua Bai\textsuperscript{1}, Peter K. Liaw\textsuperscript{6,*}, & Tongde Shen\textsuperscript{1,*}

\textsuperscript{1}Clean Nano Energy Center, State Key Laboratory of Metastable Materials Technology and Science, Yanshan University, Qinhuangdao 066004, China. \textsuperscript{2}Department of Mechanical Engineering, The University of Hong Kong, Hong Kong, 999077, China. \textsuperscript{3}Neutron Scattering Division, Oak Ridge National Laboratory, Oak Ridge, TN 37831, USA. \textsuperscript{4}State Key Laboratory of Physical Chemistry of Solid Surfaces, College of Chemistry and Chemical Engineering, Xiamen University, Xiamen 361005, China. \textsuperscript{5}State Key Laboratory for Advanced Metals and Materials, University of Science and Technology Beijing, Beijing 100083, China. \textsuperscript{6}Department of Materials Science and Engineering, The University of Tennessee, Knoxville, TN 37996, USA. \textsuperscript{7}These authors contributed equally: Kangkang Wen and Xuecheng Cai. Correspondence and requests for materials should be addressed to B.S. (email: sunbaoru@ysu.edu.cn), P.L. (email: pliaw@utk.edu), or to T.S. (email: tdshen@ysu.edu.cn).

Over 100 years, stainless steels have been extensively used as engineering materials in many areas\textsuperscript{1-4}. However, the strength-ductility trade-off\textsuperscript{1,3} and insufficient elevated-temperature strength largely hinder their processing and applications. Here, we report a novel Co-free Fe\textsubscript{47}Cr\textsubscript{16}Ni\textsubscript{26}Ti\textsubscript{6}Al\textsubscript{5} medium-entropy stainless steel (MESS) strengthened by high-density coherent L\textsubscript{12} nanoprecipitates (NPs). We use a thermodynamic approach to pursuing a large volume fraction of stable L\textsubscript{12} NPs in the coarse-grained face-centered-cubic (FCC)-structured matrix of the MESS, which is then readily fabricated through
conventional casting and thermomechanical-treatment techniques. The MESS exhibits a high ultimate tensile strength of 1.35 gigapascals (GPa) and a great total elongation of 36% at room temperature (RT), evading the strength-ductility trade-off dilemma in conventional stainless steels. The high strength is mainly due to the chemical-ordering strengthening of high-density coherent L1\textsubscript{2} NPs. The ductile L1\textsubscript{2} NPs cooperative with the dynamic refinement of the deformation substructures endow the MESS with an excellent work-hardening ability and a large uniform ductility. Furthermore, the MESS maintains a high yield strength of \( \sim 0.8 \) GPa at 700 °C, which is better than many Fe-based superalloys and stainless steels, even comparable to some Ni-based superalloys. The steady-state creep rates at 750 °C are at least two orders of magnitude lower than those of conventional Ni-based superalloys and heat-resistant steels. The excellent creep resistance is achieved via the strong interactions between sliding dislocations and stable L1\textsubscript{2} NPs at elevated temperatures, which effectively impedes the dislocation movement. The present study has huge potential for designing cost-effective engineering MESSs with excellent mechanical performance for practical applications.

Although renewable energy is on the rise, fossil fuels are likely to remain as the primary source of energy in the coming decades. Thus, it is important to use these resources as efficiently as possible, for both economic reasons and minimization of pollutants. Stainless steels containing Fe, Cr, and Ni elements have been extensively used as high-temperature materials, but they usually exhibit limited strengths at
ambient and elevated temperatures. Microstructure refinement is an effective strengthening strategy for stainless steels, whereas it comes at the expense of ductility because of the strength-ductility trade-off. Moreover, fine-grained alloys often suffer from prominent grain growth and considerable reduction in strength (i.e., thermal softening) at high-temperatures. Superalloys comprising high concentrations of Ni, Cr, Co, and Fe elements are used as engineering materials in many fields because they can withstand the high operating temperatures and stresses of components. However, they have a large number of expensive elements, such as Co, Ta, and Mo, resulting in very high production costs. On the other hand, some elements (Re and Ru) have been added to superalloys to improve creep strength. Nevertheless, these elemental additions can render alloys to possess higher density and greater cost. Furthermore, the compositions of cast superalloys have to exhibit good castability. However, the addition of heavy elements (Re, W) tends to cause density inversion and results in the formation of casting freckle defects. Therefore, the development of novel high-performance materials for elevated-temperature applications is urgently needed but is still a great challenge. Unfortunately, the conventional metallurgical-design strategies based on single-principle-element systems, in most cases by adding small amounts of alloying elements or fine-tuning the content of specific elements, have approached their limits for a substantial performance improvement. The emergence of high-entropy alloys (HEAs) and/or medium-entropy alloys (MEAs) presents an unconventional concept and approach, which have been demonstrated to be one of the most promising structural alloys with a strength-ductility synergy and/or superior
elevated-temperature performance. Nevertheless, these alloys still do not escape from the dilemma of high contents of expensive elements, and it is still challenging to develop cost-effective engineering HEAs/MEAs for wide and practical applications\textsuperscript{10-19}. The development of high-performance low-cost Co-free HEAs/MEAs is of great significance and thereby attracts much attention in recent years\textsuperscript{20-27}.

In this study, we combine a thermodynamics approach that is employed for developing compositionally complex alloys like HEAs/MEAs (see Method) with experimental screening to determine an optimum alloy composition of our Co-free MESS $[\text{Fe}_{47}\text{Cr}_{16}\text{Ni}_{26}\text{Ti}_{6}\text{Al}_{5}]$ (atomic percent, at. %), wherein the contents of Fe, Cr, and Ni are close to those of conventional stainless steels (Fig. 1a) and Fe-based superalloys. The high-resolution neutron-diffraction pattern of the MESS, as shown in the Extended Data Fig. 2a, indicates that the alloy is composed of FCC and L1\textsubscript{2} phases. An electron backscatter diffraction (EBSD) image (Fig. 1b) reveals a full recrystallized microstructure, which exhibits a uniform distribution of equi-axed grains with an average size of $70 \pm 22$ micrometre ($\mu$m) (Fig. 1d). Besides, annealing twins are occasionally observed in the matrix (Fig. 1b). The crystallography and chemical composition were further investigated, employing transmission electron microscopy (TEM). The dark-field transmission electron microscopy (DF-TEM) image (Fig. 1c) clearly reveals that a high number density of NPs is uniformly dispersed in the matrix. The average particle size is measured to be $\sim 14.4 \pm 3.1$ nanometer (nm) (Fig. 1d). The selected area electron diffraction (SAED) pattern in the inset of Fig. 1c confirms
the L$_{12}$-type precipitates, wherein superlattice spots from ordered L$_{12}$ NPs, superimposed on the fundamental reflections from the FCC matrix along the [001] direction, are clearly detected. Qualitative energy-dispersive X-ray spectroscopy mapping in the scanning-TEM mode (STEM-EDS) results are presented in the Extended Data Fig. 3a. The FCC matrix phase and the ordered L$_{12}$ NPs have distinctively different compositions. Cr is strongly partitioned to the matrix phase forming a solid solution rather than being partitioned to Fe to form a Fe-Cr (σ) phase. Meanwhile, the NPs are revealed to be enriched in Ni and Ti. From the high-angle annular dark-field STEM (HAADF-STEM) (Fig. 1e and Extended Data Figs. 3b and 3c), we observe a coherent FCC/L$_{12}$ interface with continuous crystal lattices, where the interplanar spacing of the ordered L$_{12}$ phase is very close to that of the FCC matrix phase (the lattice mismatch of ~ 0.19%). This trend agrees with the neutron-diffraction results, which suggest that the lattice mismatch between the FCC matrix and the L$_{12}$ phase is only ~ 0.124% (Extended Data Fig. 2a). Such a low lattice mismatch would effectively decrease the nucleation barrier, enabling NPs with an extremely high number density (more than $10^{22}$ m$^{-3}$) and a small particle size (~ 14.4 ± 3.1 nm). Moreover, the HAADF-STEM image (Fig. 1e) confirms that the NP has an L$_{12}$-type ordering structure with a sublattice occupied by different elements. The elements with higher atomic numbers (Ni, Fe, and Cr) take up face-centered locations of the FCC crystal lattice, whereas elements with lower atomic numbers (Ti and Al) occupy angular positions (Extended Data Fig. 3c). To further probe the L$_{12}$ NPs, we performed three-dimensional atom probe tomography (3D-APT). Atom maps (Fig. 1f)
show that Fe and Cr are enriched in the matrix whereas Ni, Al, and Ti are dominant in
the NPs. The three-dimensional morphologies of the ordered L1\textsubscript{2} NPs, and the FCC
matrix were revealed by reconstructing 60 at. % Ni and 22 at. % Cr iso-concentration
surfaces, respectively (Fig. 1f). The stacked NPs can be observed more clearly,
wherein the interconnected FCC matrix serves as a frame for the ordered L1\textsubscript{2} NPs.
The one-dimensional concentration profiles of the phase interface (Fig. 1g)
demonstrate that the contents of all alloying elements vary continuously from the FCC
matrix to the L1\textsubscript{2} NP with a diffuse interface width of $\sim 4$ nm. The one-dimensional
concentration profiles across an individual L1\textsubscript{2} NP also exhibit a long-range periodic
fluctuation without an abrupt composition change within the NP (Extended Data Fig.
4a). The chemical compositions of the FCC matrix and the L1\textsubscript{2} NPs acquired from the
flat region of the profiles are Fe\textsubscript{55.6}Ni\textsubscript{16.9}Cr\textsubscript{20.7}Al\textsubscript{2.8}Ti\textsubscript{4.0} and Ni\textsubscript{66.0}Fe\textsubscript{7.4}Cr\textsubscript{1.3}Al\textsubscript{8.8}Ti\textsubscript{16.5}
(at. %), respectively, yielding a (Ni + Fe + Cr): (Al + Ti) ratio of $\sim 3:1$ (A\textsubscript{3}B-type) for
L1\textsubscript{2} NPs. Using the lever rule analysis\textsuperscript{16}, the volume fraction of the L1\textsubscript{2} phase is
determined to be $\sim 15\%$ (Extended Data Fig. 4b), which is consistent with the volume
fraction of $\sim 17.3\%$ determined by the Rietveld refinement from neutron-diffraction
patterns (Extended Data Fig. 2a).

Figure 2a and Extended Data Fig. 5 exhibit the tensile stress-strain curves of the
MESS performed from room temperature (RT) to 800 $^\circ$C. A base alloy (Fe\textsubscript{47}Cr\textsubscript{16}Ni\textsubscript{26},
see Methods) was also tested for comparison. The MESS exhibits an extraordinary
combination of high strength and great ductility. The RT yield strength ($\sigma_\text{y}$) and
ultimate tensile strength ($\sigma_\text{u}$) of MESS are as high as 927 and 1,353 megapascals
(MPa), respectively. Note that the $\sigma_y$ of MESS is five times that (165 MPa) of the base alloy (Fig. 2a). Surprisingly, such a high strength comes with a high uniform elongation of 30% and a great total elongation ($\varepsilon$) of 36%. At such a high $\sigma_y$, the MESS still shows a strong strain-hardening ability ($\sigma_u - \sigma_y > 400$ MPa, $\sigma_y/\sigma_u < 0.7$).

According to the Considere’s criterion, homogeneous deformation can be maintained when the strain-hardening rate (SHR), $d\sigma/d\varepsilon$, is above the flow stress, $\sigma^{\text{28}}$. Thus, the higher the SHR, the greater the ductility. The higher SHR of the FeCrNiTiAl MESS - when compared with the FeCrNi base alloy - (Fig. 2b) endows it with a satisfactory homogeneous ductility (Fig. 2a). Moreover, the MESS presents a typical three-stage feature of strain hardening, which has been commonly observed in precipitation-strengthened alloys. The corresponding strain-distribution maps obtained by digital image correlations (DIC) at various global strains and at ambient temperature are exhibited in Fig. 2c. The sample shows a very diffuse strain distribution throughout the gauge section when being strained below 30% [Figs. 2(c1) - (c3)], indicating a homogenous deformation. As the deformation proceeds, necking occurs at a ~ 30% strain [a red dashed frame in Fig. 2(c4)], and the sample quickly fractures due to a significant strain localization [Fig. 2(c5)]. The MESS also exhibits superior elevated-temperature mechanical performances. Not much drop in both the yield strength and ductility but an obvious serrated response is observed between 300 and 500 °C (Extended Data Fig. 5). The $\sigma_y$ values of the MESS at 600 °C and 700 °C are still as high as 808 MPa and 802 MPa, respectively (Fig. 2a and Extended Data Fig. 5). Noteworthy, the MESS maintains a high strain-hardening rate at 600 °C (Fig.
2b), resulting in a high $\sigma_u$ of 1,074 MPa (Fig. 2d and Extended Data Fig. 5). The
dimple morphology of the fracture surface confirms the intrinsic ductile feature of the
MESS at room and elevated temperatures (Extended Data Fig. 6). The fractographic
feature changes from a transgranular fracture mode (below 600 °C, Extended Data Fig.
6a) to a mixed mode of intergranular and transgranular fracture (600 °C, Extended
Data Fig. 6b) and then to intergranular fracture (700 °C, Extended Data Fig. 6c).

To reveal the underlying mechanisms for the three-stage hardening behavior, the
evolution of the microstructures of the MESS deformed to different strains (3%, 10%,
23%, and fracture) were examined. The sharp decline of the SHR (Fig. 2b) of MESS
at the early strain stage (< 3% strain, stage 1) after yielding can be attributed to the
prevalence of dislocations re-arrangement, such as the cross-slip and annihilation of
screw dislocations of opposite signs. At a strain of ~ 3% (Fig. 3a), dislocation slip
is preferentially activated at the grain boundary, and planar-arrayed slip bands lying
essentially on {111} planes of the matrix appear. With further straining, the SHR
drops slowly (Fig. 2b). It can be clearly observed that the dislocations operated
mainly in a planar way, wherein dense {111} plane slip bands are characterized at a
strain of 10% of the MESS (Fig. 3b). The deformation mode transits from a cross slip
to planar slip, indicating that the underlying deformation starts to be dominated by the
enhanced ordering effect. Meanwhile, more dislocations are further activated in
grain interiors (Fig. 3b). Figure 3c indicates that the coherent NPs have been fully
sheared by these cutting dislocations. The inverse fast Fourier transformation (IFFT)
pattern (Fig. 3c inset image) taken near the dislocation cutting point (the upper left
black circles of Fig. 3c) reveals a profuse multiplication of edge dislocations, indicative of a remarkable strain-hardenening capability of the material. With increasing the strain to 23%, the dislocations continue operating in a planar way, forming a substructure of Taylor lattices (yellow arrows), where the dislocation motion is confined to the {111} primary slip systems (Fig. 3d). The averaged interspacing of these deformation substructures decreases from about 1 μm (slip bands at a 10% strain) to less than 200 nm (Taylor lattice at a 23% strain) as the plastic strain progresses. Such a pronounced planar slip of dislocations can be attributed to the existence of high-density coherent L12 ordered NPs, since it has been demonstrated that the shearable precipitates as well as the short-range order can significantly promote slip planarity. A similar observation has also been identified in a Fe-Mn-Al-C steel with nano-sized carbides. The continuous multiplication and intersection of the planar slip bands result in a dynamic subdivision and refinement of matrix grains during deformation. The refinement and intersection of these deformation substructures can serve as an effective carrier for dislocation accumulation, giving rise to an enhanced strain hardening, and being helpful to delay the onset of plastic instability. Thus, the slip bands refinement is the dominant mechanism for the high SHR (Fig. 2b) and high ductility of MESS, namely the slip-bands-refinement-induced plasticity (SRIP) effect. Figures 3d and f shows the deformation substructures of the fractured sample. To accommodate the large macro-strain, the accumulated in-plane dislocations of the Taylor lattice further transform into crystallographically aligned high-density dislocation walls (HDDWs).
and microbands (MBs). The diffraction vector shows that they are near parallel to the trace of the \{111\} slip plane, demonstrating that they are the substructures induced by a further refinement of slip bands\(^{13,33}\), which dominate the late-stage deformation process (nonuniform deformation) until the fracture failure occurs\(^{13}\). Note that the fully coherent relationship among the FCC matrix, L\(_{12}\) NPs, and the nanometer-scale particle size of these NPs can also contribute to the ductility of the MESS. Hence, the external force can be uniformly dispersed at coherent interfaces between the precipitates and matrix\(^{34}\), and the stress concentration can be largely alleviated, effectively inhibiting the premature nucleation of cracks.

The strengthening and ductile nature of the L\(_{12}\) NPs are well evidenced by in-situ neutron-diffraction results (Fig. 4). The behavior of lattice strain under a true stress at both RT and 600 °C (Figs. 4a and 4b) indicates that the L\(_{12}\) NPs can not only strengthen the matrix but also co-deform with it, as evidenced by the greatly increased lattice strains of L\(_{12}\)’s unique \{210\} and \{211\} diffraction peaks after yielding, i.e., the increasing rate of lattice strains, compared to those fundamental L\(_{12}/FCC\) diffraction peaks. Notably, the L\(_{12}\)’s unique \{210\} and \{211\} diffraction peaks deflect together with the fundamental diffraction peak of \{220\} (indicated by the red arrow), suggesting a co-deformation behavior between the L\(_{12}\) NPs and the FCC matrix. In addition, the plastic deformability of the precipitates can be assessed by examining the ratio of the full width at half-maximum (FWHM) to the d-spacing\(^{35}\). In this case, the greatly increased FWHM/d values of the \{210\} and \{211\} diffraction peaks of L\(_{12}\) NPs as a function of true strain (Figs. 4c and 4d) clearly demonstrate their ability to
undergo plastic deformation. Consequently, the high-density coherent and ductile L1$_2$ NPs working together with the dynamic refinement of the deformation substructures during plastic deformation endow the MESS with an excellent strain-hardening ability and a large uniform ductility. The precipitation strengthening from high-density L1$_2$ NPs together with solid-solution strengthening and dislocation hardening elevate the yield strength of the MESS to near 1 GPa (see Methods), wherein the precipitation-strengthening offers the greatest contribution (~ 60%) to the yield strength. Moreover, the precipitation-strengthening effect remains active at elevated temperatures, which can be evidenced by the plot of the lattice strain versus true stress at 600 °C (Fig. 4b). It is observed that the lattice strains of L1$_2$’s unique {210} and {211} diffraction peaks continue to increase beyond ~ 900 MPa, while the lattice strains of the fundamental diffraction peaks of FCC/ L1$_2$, e.g., {111} and {200}, begin to deflect towards smaller values. This behavior indicates that even at 600 °C, the L1$_2$ NPs continue to bear a larger load and serve as a source of strengthening. Extended Data Fig. 7 shows the deformation microstructure of the 10%-strained sample at 600 °C. Planar dislocation slips on the {111} planes are prevalent (Extended Data Fig. 7a), exhibiting a similar deformation mechanism to that at RT (Fig. 3b). The close-up view (Extended Data Fig. 7b) further reveals that the coherent L1$_2$ NPs within the planar slip bands are intensively sheared by the planar dislocations. The ordering strengthening from the densely coherent L1$_2$ NPs is supposed to remain effective at elevated temperatures, endowing the MESS with a good combination of high yield strength and reasonable
work-hardening ability at elevated temperatures up to 600 °C (Fig. 2b). In addition, previous work has indicated that L1_2 NPs can remain stable after aging at the elevated temperatures (between 525 and 600 °C). Particularly, the L1_2 NPs of our MESS grew gradually from 14.4 ± 3.1 nm to 27.7 ± 5.2 nm and maintained a spherical morphology after a high-temperature treatment at 700 °C for 200 hours (h) (Extended Data Figs. 8a and b). The superior thermal and structure stability can be mainly ascribed to the coherent FCC/L1_2 interface, which sustains a small lattice mismatch (0.42%) at 700 °C (Extended Data Fig. 8c). However, the ductility remarkably decreases at 700 °C. Such a degradation of ductility is generally attributed to the precipitation of the brittle L2_1 phase at grain boundaries (Extended Data Fig. 9), which can account mainly for the transition from the intragranular to intergranular fracture (Extended Data Fig. 6). Nevertheless, the L2_1 phase was not well captured by the neutron-diffraction measurements at both RT and 600 °C. Instead, only a very minor L2_1’s 220 peak was observed (Extended Data Figs. 2a and 2b), indicating a very low content, which may not seriously deteriorate the ductility of the MESS at temperatures below 700 °C. Similar embrittling behaviors have also been frequently observed in many commercial Ni-based superalloys, such as the U720Li, Waspaloy, GH4033, etc.

Figure 5a shows a comparison of RT-mechanical properties of the MESS with those of commercial stainless steels, Fe-based superalloys, and Co-free Fe-, and Ni-based HEAs/MEAs. The present MESS exhibits yield strength two to four times that of austenitic-stainless steels and a higher product of σ and ε^3. Besides, the product of
\( \sigma_u \) and \( \varepsilon \) of our MESS (~ 50 GPa \( \cdot \) %) is four to five times those of precipitation-hardening (PH) stainless steels with comparable strengths\(^3\). Moreover, compared to the body-centered-cubic (BCC)-structured stainless steels [ferritic (F), martensitic (M), and M-F duplex], our MESS displays a two to three times yield strength and product of \( \sigma_u \) and \( \varepsilon \)\(^3\). Even when compared with the Fe-based superalloys\(^2\) and recently reported Co-free Fe- and Ni-based HEAs/MEAs\(^{20-27}\), our MESS also displays a higher strength-ductility combination. The exceptional combination of the high strength and large ductility distinguishes our MESS from other commercial stainless steels, Fe-based superalloys, and most Co-free Fe-, and Ni-based HEAs/MEAs. We also compare the elevated-temperature strength of our MESS with some conventional Fe-based superalloys and Co-free HEAs/MEAs (Fig. 5b)\(^{2,22,39-41}\). Almost no decay in the high yield strength (~ 800 MPa) of the MESS can be observed at temperatures below 700 °C. This high yield strength well exceeds those of most Fe-based superalloys and Co-free HEAs/MEAs. Even at a high temperature of 800 °C, our MESS still has a relatively high yield strength of ~ 600 MPa. The creep resistance of our MESS was also evaluated.

High creep resistance is required for materials used at high temperatures. Conventional ferritic and austenitic heat-resistant stainless steels, however, do not have sufficient creep resistance in harsh environments\(^1\). The \( \gamma \) prime phase-precipitation strengthened Ni-based superalloys exhibit good creep strength and corrosion resistance at high temperatures of 700 °C and above. Nevertheless, these Ni-based superalloys are excessively expensive due to the high content of Co (10 - 20
weight percent, wt. %) and/or Mo and W (6 - 8 wt. %). We performed tensile creep
tests of our low-cost MESS at a high temperature of 750 °C. Figure 5c shows the
creep strain versus time curves at different applied stresses. The creep strain is
observed to increase with the applied stress, whereas the MESS crept at 100 MPa
shows a small creep strain of less than 4% after 300 h. This is a very demanding
creep-test condition, where conventional alloys, such as a type 347 stainless steel
ruptured within ~ 100 to 300 h. We further calculated the steady-state creep rate at
different applied stresses, as displayed in Fig. 5d. At the higher stress level (750
°C/300 MPa), the steady-state creep rate of the MESS is 4.89 × 10^{-7} s^{-1}, which is at
least 3 orders of magnitude lower than those of conventional Ni-based superalloys,
such as Haynes 282 (6.4 × 10^{-4} s^{-1} at 760 °C/290 MPa), Inconel 740 (4.1 × 10^{-4} s^{-1}
at 750 °C/300 MPa), and Sanicro 25 (1.46 × 10^{-3} s^{-1} at 750 °C/240 MPa).
The deformation-microstructure analysis was performed in creep-strained specimens
in order to explore the interactions between dislocations and L1_2 NPs. The bright-field
(BF) TEM image (Extend Data Fig. 10a) shows dislocation configurations of the alloy
crept at 750 °C/70 MPa for 300 h. Most of the dislocations are observed to be strongly
curved, revealing an effective impediment of NPs on dislocation movement. The inset
of Extend Data Fig. 10a presents the interaction details, where dislocation climbing up
the L1_2 NPs can be clearly identified. The dislocation climbing is principally
accommodated by the long-range diffusion of vacancies, retarding dislocations to
overcome obstacles. A low steady-state creep rate (~ 1.71 × 10^{-8} s^{-1}) is thus obtained
during creep deformation under a relatively small, applied stress of 70 MPa.
Meanwhile, some discrete dislocation loops can also be observed, as indicated by the yellow arrows in Extend Data Fig. 10a, indicating that the Orowan bypassing process is activated. The size-dependent critical resolved shear stress (CRSS) for Orowan looping can be estimated as \( \tau_c = Gb/l \) where \( \tau_c \) is the CRSS, \( G \) is the shear modulus, \( b \) is the Burgers vector, and \( l \) is the inter-particle spacing. Assuming that the volume fraction of the particle remains constant, the average inter-particle spacing, \( l \), increases with precipitates coarsening. It is noted that particle ripening would inevitably occur to some extent in the later stage of the high-temperature creep, leading to a decrease of the Orowan shear stress and dislocation looping around the coarser precipitates. The BF-TEM image (Extend Data Fig. 10b) shows dislocation configurations of the alloy crept at 750 °C/150 MPa for 128 h, where prevalent dislocation shearing through L1_2 NPs can be clearly observed. It can be inferred that the increased applied stress (150 MPa) enables dislocations to overcome the shear resistance of L1_2 NPs, resulting in a slip-plastic deformation. The dislocation shearing is identified as the dominant deformation mechanism during the creep process at 750 °C/150 MPa under the present circumstances. In either case, the interactions between dislocations and L1_2 NPs can impede the movement of dislocations, thus effectively enhancing the creep resistance of MESS.

Extended Data Fig. 11a shows the comparison between the ultimate tensile strength (UTS) and the raw material costs for typical 304 austenitic-stainless steels, Fe-based superalloys\(^2,4\), some recently reported HEAs/MEAs\(^{13,18,24,47-50}\), and the present MESS. As can be seen, our MESS not only produces impressive mechanical properties, but
also has a raw material cost lower than most of the counterparts. Moreover, we have
also used the parameter of the ratio of UTS to the raw material costs to further
manifest the strengthening efficiency of a promising material. As can be seen from the
Extended Data Fig. 11b, our MESS shows a great advantage in potential engineering
applications.

In conclusion, we have developed a novel MESS with an excellent strength-ductility
synergy over a wide temperature range, which can be fabricated readily through
conventional casting and thermomechanical treatment techniques. Our material design
principle has huge potential for fabricating cost-effective engineering MESS with
exceptional mechanical performance to replace, at least partially, both conventional
low-strength stainless steels and expensive nickel-based superalloys that are widely
utilized worldwide.


17. Pan, Q. *et al.* Gradient cell-structured high-entropy alloy with exceptional strength and


Tensile properties of an AlCrCuNiFeCo high-entropy alloy in as-cast and wrought conditions.


40. Daoud, H. M., Manzoni, A. M., Wanderka, N. & Glatzel, U. High-Temperature Tensile Strength of Al$_{10}$Co$_{25}$Cr$_{8}$Fe$_{15}$Ni$_{36}$Ti$_{6}$ Compositionally Complex Alloy (High-Entropy Alloy).


41. Jiang, W., Yuan, S., Cao, Y., Zhang, Y. & Zhao, Y. Mechanical properties and deformation mechanisms of a Ni$_2$Co$_1$Fe$_1$V$_{0.5}$Mo$_{0.2}$ medium-entropy alloy at elevated temperatures. *Acta. Mater.* **213** (2021).


47. He, J. Y. *et al.* A precipitation-hardened high-entropy alloy with outstanding tensile properties.


48. Liang, Y. J. *et al.* High-content ductile coherent nanoprecipitates achieve ultrastrong


Fig. 1 | Conceptual design and microstructural characterizations of the MESS. a Position of our alloy in the Fe-Cr-Ni phase diagram falling within the composition range of stainless steels. b EBSD image of the MESS with equiaxed grains. c DF-TEM image of the MESS showing the high-density NPs. The SAED pattern in the inset verifying the L1\textsubscript{2}-type of the NPs. d Statistical distributions for the average size of the FCC matrix grains and L1\textsubscript{2} NPs (d denotes the average size). e High-resolution STEM image illustrating the interfacial coherency of the FCC matrix/L1\textsubscript{2} NP (Z. A. denotes the zone axis). f Representative atom maps collected from the MESS, using the 3D-APT showing the distribution of each element. Fe and Cr are enriched in the matrix, whereas Ni, Al, and Ti are enriched in the NPs, and three-dimensional reconstruction of 60 at. % Ni and 22 at. % Cr iso-concentration surfaces presenting the morphologies of the ordered L1\textsubscript{2} NPs and the FCC matrix. g One-dimensional concentration profiles showing the elemental distributions from the matrix to the NP.
**Fig. 2 | Exceptional strength-ductility combination of the MESS.**

a Uniaxial engineering tensile stress-strain curves at different temperatures. b Strain-hardening rate versus true strain. The intersections with the true stress–strain curves are marked with arrows to indicate the onset of necking instability. C Strain-field distribution of the MESS during tensile testing at ambient temperature (field of view: 5.2 mm × 2.2 mm). Evolution of micro-strains along the loading direction at different macro-strains: 3% (c1), 10% (c2), 23% (c3), 30% (c4), and 36% (c5). D Variations of $\sigma_y$, $\sigma_u$, and elongation of the MESS with testing temperature.
Fig. 3 | Deformation microstructures of the MESS at RT. a Deformation microstructure of the 3%-strained sample. Grain boundary (yellow arrow) emits dislocations, and a small amount of slip trace (white dashed line) occurs in the form of the planar dislocation slip. b Activations of more \{111\} slip traces (white dashed line) at the 10% strain. c A close-up view of the region marked by the yellow rectangle in b revealing that dislocations shear through L1₂ NPs. The IFFT image in the inset showing the profuse multiplication of edge dislocations (in yellow dashed circles) near the dislocation cutting point (inside the upper left white circle). d High-density \{111\} slip traces leading to the formation of Taylor lattices (yellow arrow) at the 23% strain. e, f Deformation microstructures of the fractured MESS. At high strains, HDDWs (e) and MBs (f) were formed. The slip trace shows that the MBs are parallel to the trace of the \{111\} glide plane (white dashed line).
Fig. 4 | In-situ neutron-diffraction results. a and b Lattice strain as a function of true stress during tensile deformation at RT and 600 °C, respectively, showing the load transfer from the soft matrix to the hard L1\textsubscript{2} NPs, a reflection of the precipitation strengthening. c and d Evolution of FWHM/d as a function of true strain, indicating the ductile nature of the multicomponent L1\textsubscript{2} NPs.
Fig. 5 | Mechanical properties of our MESS compared with other alloys. a Yield strength versus the product of the ultimate tensile strength and ductility of the MESS, compared with those of stainless steels, Fe-based superalloys, and some Co-free HEAs/MEAs. b Variation of yield strength with testing temperature of the MESS, compared with those of conventional stainless steels, Fe-based superalloys, and some HEAs/MEAs. There, of course, are a few advanced Ni-based superalloys and stainless steels not shown for comparison, due to their difficulty to achieve mass industrial production (for example, additive manufacturing, high-pressure torsion, and liquid-nitrogen cold rolling). Some high Co and, Ni content (more than 50 at. %) HEAs/MEAs are not shown for comparison because of expensive costs, as well. c Creep strain versus time curves of the MESS at 750 °C under different applied stresses, the arrows denote the that samples are not fractured during creep testing. d Creep rate versus applied stress curves of the MESS and some other conventional Ni-based superalloys and heat-resistant steels.
Methods

Compositional design and phase calculation. The ternary FeCrNi alloy with an FCC structure was initially chosen as the base alloy, which was then alloyed with Ti and Al to generate L1₂-type coherent NPs. Three main criteria were considered in the alloy-design process. First, a high content of Cr (~16 at. %) - close to that of the 304 stainless steel - is selected in the base alloy. The high content of Cr can promote the phase separation and reduce the difference in the lattice parameters between the FCC matrix and L1₂ NPs, thereby stabilizing the coherent interfaces and lowering the driving force for competitive coarsening. Additionally, a high content of Cr can also render the MESS a superior corrosion and oxidation resistance. Second, a high content of Ni is expected, which can facilitate the precipitation of L1₂ NPs as much as possible. However, the Ni content should be kept as low as possible to promote the wide applicability of our MESS. The Ni content in our MESS is thus ideally controlled within 28 at. % - which is the maximum amount of Ni in stainless steels - to ensure a comparable material cost to commercial stainless steels. Third, L1₂ NPs should remain as stable as possible at high temperatures, while inhibiting the formation of other brittle intermetallic compounds in MESS. Under the circumstances, the addition of Al and Ti should be well controlled to ensure a stabilized matrix phase without excess complex phases.

The empirical criteria, $\Omega = T_m \Delta S_{mix}/\Delta H_{mix} \geq 1.1$ and $\delta \leq 6.6\%$, - which have been widely used to predict a stable solid-solution matrix in HEAs - are utilized to optimize the contents of Al and Ti in our MESS. Meanwhile, $\Omega$ should approach the threshold
value of 1.1, where the formed secondary phases may be more beneficial for mechanical performance (i.e., coherent precipitates). Herein the parameters, $\Delta H_{\text{mix}}$ (enthalpy of mixing), $\Delta S_{\text{mix}}$ (entropy of mixing), $\delta$ (atomic size difference), and $T_m$ are defined as follows\textsuperscript{11,54}

\begin{equation}
\Delta H_{\text{mix}} = \sum_{i=1, i \neq j}^{n} \Omega_{ij} c_i c_j = \sum_{i=1, i \neq j}^{n} 4\Delta H_{ij}^{\text{mix}} c_i c_j 
\end{equation}

\begin{equation}
\Delta S_{\text{mix}} = -R \sum_{i=1}^{n} c_i \ln c_i = \sum_{i=1}^{n} c_i = 1
\end{equation}

\begin{equation}
\delta = \sqrt{\sum_{i=1}^{n} c_i (1 - r_i/\bar{r})^2}, \bar{r} = \sum_{i=1}^{n} c_i r_i
\end{equation}

\begin{equation}
T_m = \sum_{i=1}^{n} c_i (T_m)_i
\end{equation}

where $(T_m)_i$ is the melting point of the $i$th component and $R$ represents the gas constant (8.314 J·K\textsuperscript{-1}·mol\textsuperscript{-1}); $\Delta H_{ij}^{\text{mix}}$ is the enthalpy of mixing of the $i$th and $j$th components based on the Miedema macroscopic model\textsuperscript{55}. In addition, $c_i$ and $c_j$ are the atomic percentages of the $i$th and $j$th constituent elements, respectively; and $r_i$ indicates the atomic radius of the $i$th component\textsuperscript{11}. On this basis, we developed three MESSs with different Ti/Al contents, i.e., Ti\textsubscript{4}Al\textsubscript{3}, Ti\textsubscript{6}Al\textsubscript{5}, and Ti\textsubscript{7}Al\textsubscript{7}. Then we first calculated equilibrium phases of the three MESSs above, as shown in Extended Data Figs. 1a, b, and c. The calculations of phase equilibria were performed with JMatPro (www.sentesoftware.co.uk), which can be used to guide the subsequent heat-treatment process.

Based on the calculation results, the homogenization and recrystallization temperatures should be controlled below 1,200 °C, above which the alloys start to melt. In addition, it is noted that the volume fraction of the harmful L2\textsubscript{1} phase decreases with the temperature in the high-temperature range of ~ 800 - 1,200 °C, and
thus, the homogenization and recrystallization temperatures selected should be as close to 1,200 °C as possible. Below 800 - 900 °C, the favorable L1₂ phase is in equilibrium with the FCC matrix phase, and its volume fraction increases with decreasing temperature, while the harmful L2₁ phase is found to decrease with decreasing temperature. However, undesired NiAl or η phases also tend to form at low temperatures, especially for the alloy with a high Ti/Al content (i.e., Ti₇Al₇) wherein the volume fraction of the NiAl phase would increase with decreasing temperature. By weighing the favorable L1₂ and other undesired phases, the aging temperature was prudently selected around 600 °C. It is also worth noting that the formation of the L1₂ phase is generally accompanied by the precipitation of σ and BCC complex phases, whereas neither of them has been observed experimentally, probably due to the incoherence of the precipitates/matrix interfaces and the corresponding large nucleation barrier.

Through the above analysis, we selected a homogenization and recrystallization temperature of 1,150 °C and a preliminary aging temperature range of 550 - 650 °C. The optimal aging temperature was selected to be 600 °C, which was determined by comparing the mechanical properties of alloys aged at different temperatures (550 - 650 °C). The tensile properties of the three alloys with different Ti/Al contents prepared by the above optimized heat-treatment processes are shown in the Extended Data Figure 1d. In order to avoid discrepancies between the actual and nominal compositions, we simultaneously performed mechanical-performance tests on two other alloys with a similar composition to Ti₆Al₅, i.e., Ti₆Al₆ and Ti₅Al₅, which are
produced by the same heat-treatment processes. As can be seen from the Extended
Data Fig. 1d, the Ti₆Al₅ alloy exhibits the best synergy of strength and ductility
among the five MESSs. Finally, combining the thermodynamics approach with the
experimental screening, we successfully determined an optimum chemical
composition of our Co-free MESS, i.e., Fe₄₇Cr₁₆Ni₂₆Ti₆Al₅ (at. %), which contains a
large number of ordered coherent L₁₂-type Ni₃(Ti, Al) NPs in the FCC-structured
FeCrNi base alloy.

**Materials preparation.** Two alloys, Fe₄₇Cr₁₆Ni₂₆Ti₆Al₅ and Fe₄₇Cr₁₆Ni₂₆ (at. %),
were fabricated by arc melting pure elements under a Ti-gettered high-purity argon
atmosphere. The starting elemental materials were at least 99.99 wt. % pure. All the
alloy ingots were repeatedly melted for at least six times to ensure a chemical
homogeneity, and finally drop-cast into a 60 mm × 20 mm × 5 mm copper mold. The
ingots were homogenized at 1,150 °C for 2 h, water-quenched to RT, and cold rolled
with a total reduction of 70% at RT. The cold-rolled sheets were recrystallized at
1,150 °C for about 1 min., and then furnace-cooled to ambient temperature. Finally,
the sheets were aged at 600 °C for 1 h and then cooled to ambient temperature by
quenching into water. Some specimens were further heat treated at 700 °C for 200 h to
evaluate the microstructural stability. The heating rate of the above heat treatments is
2 °C min⁻¹, and all the above heat treatment processes were carried out under a
vacuum condition (less than 0.001 MPa). The specimens for subsequent tests were cut
by electrical discharge machining.

**Comparison of raw material cost.** The alloys’ cost was estimated using the
price of a pure element, which is available at www.shfe.com.cn. Thus, the estimated
cost may be different from the market price.

**Microstructural characterization.** The matrix grains and fracture
morphologies of specimens were examined, using a scanning electron microscope
(SEM) and electron backscatter diffraction (EBSD, FEI helios G4 CX). The
microstructures of specimens were characterized by employing a transmission
electronic microscope (TEM, FEI Talos F200X), which was operated at 200 kV and
equipped with energy dispersive spectroscopy (EDS). The NPs were further
characterized by an aberration-corrected TEM (FEI Themis Z) operated at 300 kV.
Specimens for the EBSD and TEM observations were made by first mechanically
grinding to a thickness of ~ 50 μm via SiC papers, then punching into discs with a
diameter of 3 mm, and finally electropolishing in an electrolytic solution containing
10 volume percent (vol. %) perchloric acid and 90 vol. % alcohol under a voltage of
26 V. X-ray diffraction (XRD) examination was carried out on a Rigaku
D/max-2500/PC X-ray diffractometer with Cu-Kα radiation (λ = 0.154 nm). The θ -
2θ scanning was conducted in the range of 30° - 100° with a scanning speed of 2°
min⁻¹. Three-dimensional atom probe tomography (3D-APT) was performed in a
CAMECA Instruments LEAP 5000XR local electrode atom probe. The specimens
were analyzed in a voltage mode, at a specimen temperature of 50 K, a pulse
repetition rate of 200 kHz, a pulse fraction of 0.2, and an ion collection rate of 0.5%
ions per field-evaporation pulse. Needle-shaped specimens required for APT were
fabricated by lift-outs and annular milling in an FEI Scios focused ion beam/scanning
electron microscope (FIB/SEM). Imago Visualization and Analysis Software version 3.8.10 (www.atomprobe.com) was employed for three-dimensional reconstructions and data analysis. The obtained compositions were used to evaluate the volume fractions of the FCC and L1$_2$ phases, using the level rule. The equation$^{16}$ can be described as:

$$f_{L1_2} = \left( c_{i_{\text{nominal}}} - c_{i_{\text{matrix}}} \right) / \left( c_{i_{\text{precipitate}}} - c_{i_{\text{matrix}}} \right),$$

where $c_{i_{\text{nominal}}}$, $c_{i_{\text{matrix}}}$, and $c_{i_{\text{precipitate}}}$ refer to the atomic ratio of each element, $i$, in the nominal alloy composition, FCC matrix, and L1$_2$ phases, respectively. Bring the atomic ratio for each elemental to the above equation, respectively, then fitting the all points to a linear relationship yields a slope, which is equal to the volume fraction of the L1$_2$ NPs.

**Mechanical tests.** Flat dog bone-shaped tensile and creep specimens with a gauge length of 5 mm and a cross-section area of 1.8 mm $\times$ 1.5 mm were cut by electrical discharge machining and polished with 2,000-grit SiC papers. A computer-controlled WDW-50S MTS testing machine was employed to investigate the tensile properties. All tensile tests were conducted at a nominal strain rate of 0.001 s$^{-1}$. For high-temperature tensile tests, specimens were first heated to the desired testing temperatures at a rate of 40 °C min$^{-1}$ and then remained at the testing temperatures for 5 min. before tensile tests. Each sample was tested three times to ensure repeatability. The tensile-loading direction was parallel to the rolling direction. A contactless strain gauge based on a digital image correlation (DIC) technique with a high accuracy of 6 μm was designed to instantaneously capture and analyze the strain images. The DIC technique, which correlates the speckle pattern between images, can numerically evaluate the strain distribution and calculate the axial strain of the tensile specimens.
The strain field was analyzed, employing a MERCURY software (www.testingequipmentie.com). The necking point and the uniform elongation were determined by the point of a maximum load. The work-hardening rate, \( \theta \), was derived by differentiating the true stress, \( \sigma_t \), over the true strain, \( \varepsilon_t \), using the equation, \( \theta = \frac{d\sigma_t}{d\varepsilon_t} \). Tensile creep tests were performed in a uniaxial tensile mode on an RDL-50 testing machine at 750 °C under different applied stresses ranging from 70 to 300 MPa in air. The creep strain was measured continuously, using a grating transducer extensometer with a displacement resolution of ± 1 \( \mu \)m. The steady-state creep rate was determined by linearly fitting the creep strain versus time curve in the steady-state creep stage.

**Neutron diffraction.** Neutron diffraction was performed on the engineering materials diffractometer, VULCAN, at the Spallation Neutron Source, Oak Ridge National Laboratory. A high-resolution neutron-diffraction mode with a d-spacing coverage of 0.5 - 3.5 Å was used to measure the undeformed MESS sample. Rietveld refinement was performed, using GSAS with EXPGUI. The MESS samples underwent tensile tests at RT and 600 °C were subjected to in-situ neutron diffraction with a high-intensity mode. The FCC and L\(_1\)\(_2\) structures were used to refine the lattice parameters (\( a_{FCC} = 0.3606 \) nm, and \( a_{L12} = 0.3611 \) nm), phase fraction (\( f_{L12} = 17.3\% \)), and lattice mismatch (0.124%) by Rietveld refinement. The full width at half-maximum (FWHM) for L\(_1\)\(_2\)’s \{210\} and \{211\} diffraction peaks was used for quantifying deformability, which was obtained through correcting the instrumental broadening with a near-perfect (broadening-free) Si sample.
Estimation of strengthening by various mechanisms. The RT yield strength ($\sigma_Y$) of our MESS is estimated based on the individual strengthening contribution, expressed as:

$$\sigma_Y = \sigma_0 + \sigma_s + \sigma_g + \sigma_d + \sigma_p$$

(5)

where $\sigma_0$ is the intrinsic strength, or the so-called lattice-friction strength, $\sigma_s$ is the solid-solution strengthening, $\sigma_g$ is the grain-boundary strengthening, $\sigma_d$ is the dislocation strengthening, and $\sigma_p$ is the precipitation strengthening. Here, the intrinsic lattice friction stress (165 ± 5 MPa) of the base Fe$_{47}$Cr$_{16}$Ni$_{26}$ alloy is used.

The present MESS can be simply treated as a FeCrNi-solvent matrix containing Ti and Al solutes, and a standard model for substitutional solid-solution strengthening, based on dislocation-solute elastic interactions, can be directly applied to evaluate the solution strengthening term, $\sigma_s$, caused by Ti and Al:

$$\sigma_s = M G c^{1.5} \sigma^0.5 / 700$$

(6)

where $G$ is the shear modulus for the MESS (81 GPa)$^2$, $c$ is the total molar ratio of Ti + Al in the simple FCC matrix (Extended Data Table 1), and $M = 3.06$ is the Taylor factor. The strength increment caused by solid-solution hardening, $\sigma_s$, is calculated to be $22 \pm 2$ MPa.

The grain-boundary strengthening contribution, $\sigma_g$, is estimated on the basis of the Hall-Petch relation:

$$\sigma_g = k_y (d_a^{0.5} - d_c^{0.5})$$

(7)

where $d_a$ and $d_c$ represent the grain size of the aged and as-cast MESS, respectively.

Using the value of $k_y (966 \pm 25$ MPa $\mu$m$^{0.5}$) derived from the FeCrNi system$^{57}$ and the
grain sizes ($d_a = 70 \mu m$ and $d_c = 249 \mu m$) measured by EBSD. One obtains $\sigma_g = 54 \pm 1$ MPa.

The strengthening contribution from dislocations ($\sigma_d$) is calculated according to the Taylor-hardening law\(^{28}\)

$$\sigma_d = MaGb\rho^{0.5}$$  \hspace{1cm} (8)

where $\alpha$ is a constant (0.2), $b$ is the magnitude of the Burgers’ vector, and $\rho$ is the dislocation density. Here, $b = \sqrt{2}/2a_{\text{MESS}}$, and the lattice parameter, $a_{\text{MESS}}$, is 0.3607 nm (measured from neutron diffraction). The dislocation density ($\rho$) is calculated by the expression of $\rho = \sqrt{2}\varepsilon/(2db)$, where $\varepsilon$ (0.104%) is the microstrain estimated from the XRD result, using the well-known Williamson-Hall method\(^{58}\), $d$ is the average grain size. Hence, $\rho = 1.88 \times 10^{13}$ m\(^{-2}\). Therefore, $\sigma_d$ is 57 ± 33 MPa.

For an alloy containing high-density NPs, the precipitation hardening, $\sigma_p$, is primarily responsible for the pronounced increase in the yield strength\(^{52, 53}\). In the present MESS, the coherent NPs are significantly sheared by dislocations after yielding (Fig. 4d). In this case, three contributing factors are considered for the yield strength, i.e., the particle-matrix coherency strengthening ($\sigma_{cs}$), modulus-mismatch strengthening ($\sigma_{ms}$), and atomic-ordering strengthening ($\sigma_{os}$)\(^{28, 47, 59}\), which can be, respectively, estimated by

$$\sigma_{cs} = M\alpha \varepsilon G(G\varepsilon)^{1.5}[rf/(0.5Gb)]^{0.5}$$  \hspace{1cm} (9)

$$\sigma_{ms} = 0.0055M(\Delta G)^{1.5}(2f/G)^{0.5}(r/b)^{3m/2-1}$$  \hspace{1cm} (10)

$$\sigma_{os} = 0.81M[\gamma_{\text{APB}}/(2b)](3\pi f/8)^{0.5}$$  \hspace{1cm} (11)

where $\alpha = 2.6$ for the FCC structure, $m = 0.85$, $\varepsilon \approx 3 \Delta a/a$ is the constrained
lattice-parameter mismatch, with $\Delta a/a = 0.0014$ in the present work, where $\Delta a$ is the difference of lattice constants between the NPs and the FCC matrix calculated from the neutron-diffraction results; $\Delta G$ is the shear-modulus mismatch between the NP’s and matrix, $\Delta G = 4$ GPa$^{47}$, $r$ is the radius (7.2 nm), and $f$ is the volume fraction ($\sim 0.17$) of the NPs. $\gamma_{\text{APB}}$ is the antiphase boundary (APB) energy of the NPs, which is strongly dependent on the Ti/Al ratio of the NPs and can be estimated to be about 0.206 J⋅m$^{-2}$ $^{36}$. The estimated strengthening contributions from the order strengthening, coherency strengthening, and modulus-mismatch strengthening are 444, 106, and 21 MPa, respectively. The first two ($\sigma_{\text{cs}}$ and $\sigma_{\text{ms}}$) occur before dislocations shear precipitates, while the latter ($\sigma_{\text{os}}$) takes place during shearing. In this case, the larger one between ($\sigma_{\text{cs}} + \sigma_{\text{ms}}$) and $\sigma_{\text{os}}$ determines the total strength increment of the shear mechanism$^{47}$. Thus, the overall precipitation strengthening from the NPs is $\sim 444 \pm 32$ MPa [$\sigma_{\text{cs}} (106 \text{ MPa}) + \sigma_{\text{ms}} (21 \text{ MPa}) = 127 \text{ MPa}$; and $\sigma_{\text{os}} = 444 \text{ MPa}$].

The estimated $\sigma_y$ of the MESS is 742 ± 66 MPa. It is apparent that the precipitation strengthening from the NPs offers the largest contribution ($\sim 60\%$) to the yield strength of the MESS. Note that further increasing the Ti/Al addition (the Extended Data Fig. 1) - which may increase the number density of NPs - would continue to increase the strength of the MESS, but inevitably at the cost of ductility. Thus, the content of Ti/Al should be tailored to achieve the best combination of strength and ductility. In addition, it is noted that the estimated $\sigma_y$ differs from the experimental value (927 MPa) to some extent. First, several intrinsic parameters (e.g., $\Delta G$, $k_y$, and $G$) are taken from other alloys in our calculations. Second, the strength...
contribution from the L2$_1$ phase at the grain boundaries is neglected. In addition, there may be some errors in data fittings, statistical values, and elemental content determinations. Nonetheless, in any case, the precipitation strengthening from the high-density coherent L1$_2$ NPs dominates the overall strength of the MESS.

Data availability

The data that support the findings of this study are available from the corresponding authors upon reasonable request.


Acknowledgements

The present work was financially supported by the National Natural Science Foundation of China (Nos. 51971195 and 11935004), the Natural Science Foundation of Hebei Province (No. B2020203037), the Youth Fund Project of Science and Technology Research of Hebei Province (No. QN2020210). P.K.L. appreciates the supports from (1) the National Science Foundation (DMR-1611180, 1809640, and 2226508) and (2) the US Army Research Office (W911NF-13–1-0438 and W911NF-19–2-0049). R.F. thanks for the support from the Materials and Engineering Initiative at the Spallation Neutron Source (SNS), Oak Ridge National Laboratory (ORNL). A portion of the current research used resources at SNS, a U.S. Department of Energy (DOE) Office of Science User Facility operated by the ORNL. The authors would like to thank Dr. Dunji Yu for his assistance in neutron diffraction experiments, and Dr. Zuohua Wang and Dr. Tingting Yang for their assistance in the TEM characterization.

Author contributions

analyses. H.Z., and Y.W. conducted correlative 3D-ATP. K.W., X.C., T.S., and P.L. analyzed the data and discussed the results. R.F. and K.A. performed the in situ neutron-diffraction experiments and analyzed the resultant data. K.W., X.C., R.F., P.L., and T.S. wrote the manuscript. All authors discussed the results and commented on the manuscript.

Competing interests: The authors declare no competing interests.

Additional information

Supplementary information is available for this paper.

Correspondence and requests for materials should be addressed to Baoru Sun, Peter K. Liaw, or Tongde Shen.

Reprints and permissions information is available at www.nature.com/reprints.
Extended Data Fig. 1 | a–c Equilibrium phase diagrams calculated by the JMatPro software for MESSs with different compositions, Ti₄Al₃ (a), Ti₆Al₅ (b), and Ti₇Al₇ (c). d Tensile curves of alloys with different Ti/Al contents.
Extended Data Fig. 2 | a High-resolution neutron-diffraction pattern of the undeformed MESS at RT, showing the co-existence of the FCC matrix ($a_{FCC} = 0.3606$ nm) and L1$_2$ phase ($a_{L12} = 0.3611$ nm) with a lattice mismatch of 0.124%. Black crosses indicating experimental observations, red lines representing the calculated fit by Rietveld refinement. b Neutron-diffraction patterns at selected tensile strains and at 600 °C, suggesting that no other phases form during tensile deformation.
Extended Data Fig. 3 | a HAADF image and STEM-EDS mappings of the MESS. The elemental mappings illustrating that Fe and Cr partition to the FCC matrix, and Ni and Ti partition to the L1$_2$ phase. b Atomic-resolution HAADF-STEM image taken from the [001] zone axis exhibiting a coherent interface between the L1$_2$ NP and FCC matrix. c A close-up view of the region marked by the white dashed rectangle in b revealing the sublattice occupations of different elements in the L1$_2$ NP and FCC matrix.
Extended Data Fig. 4 | a One-dimensional concentration profiles from the APT results showing the elemental distributions. b Lever-rule diagram of the chemical composition of the MESS obtained from Fig. 2b indicating the volume fraction of the L1₂ NPs in the MESS through the slope of the line.
Extended Data Fig. 5 | Uniaxial tensile stress-strain curves of the MESS at elevated temperatures.
Extended Data Fig. 6 | SEM micrographs of the fracture surface of the MESS deformed at RT (a), 600 °C (b), and 700 °C (c). Jagged lines on the fracture surfaces of 600 °C and 700 °C (especially 700 °C) clearly indicating that the sample fractured in an intergranular manner, i.e., along the grain boundaries.
Extended Data Fig. 7 | **a** Deformation microstructures of the 10%-strained sample at 600 °C showing prevalent planar-dislocation slips on the \{111\} planes. **b** A close-up view of the region marked by the black rectangle in **a** revealing dislocations shearing through L1₂ NPs.
Extended Data Fig. 8 | NPs of the MESS annealed at 700 °C for 200 h. a HAADF-TEM image. Inset showing the corresponding SAED pattern. b Statistical distributions of the particle sizes. c High-resolution-TEM (HRTEM) image illustrating the interfacial coherency of the FCC matrix/L1$_2$ NP after ageing at 700 °C for 200 h, d(111)$_{L1_2}$ = 0.2158 nm, and d(111)$_{FCC}$ = 0.2149 nm, resulting in a small lattice mismatch of 0.42%.
**Extended Data Fig. 9** | Microstructural characterization of the grain-boundary L$_2^1$ phase.

Elemental mappings illustrating that Ni, Al, and Ti partition to the L$_2^1$ phase. Inset in the HAADF image showing the indexed convergent beam electron-diffraction pattern of the L$_2^1$ phase with a BCC structure.
Extended Data Fig. 10 | TEM characterization of the deformation microstructures of the creep-strained specimens. a BF-TEM image of the alloy crept at 750 °C/70 MPa for 300 h, most of dislocations being strongly curved. Inset showing the detailed interactions where a sliding dislocation is climbing up the encountered L12 NPs. b BF-TEM image of the alloy crept at 750 °C/150 MPa for 128 h, prevalent dislocations shearing through L12 NPs (yellow arrows).
**Extended Data Fig. 11** | a Comparison between the ultimate tensile strength and raw material cost for typical 304 austenitic stainless steels, Fe-based superalloys, some recently reported HEAs/MEAs, and the present MESS. 

b Comparison between the yield strength and ratio of the ultimate tensile strength (UTS) to raw material cost for typical 304 austenitic-stainless steels, Fe-based superalloys, some recently reported HEAs/MEAs, and the present MESS.
**Extended Data Table 1** | Chemical compositions of the FCC matrix and L1\textsubscript{2} phase in the MESS characterized by the 3D-APT measurement.

<table>
<thead>
<tr>
<th>Phase</th>
<th>Chemical composition (atomic percent, at. %)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Fe</td>
</tr>
<tr>
<td>Matrix</td>
<td>55.6 ± 0.5</td>
</tr>
<tr>
<td>L1\textsubscript{2}</td>
<td>7.4 ± 0.8</td>
</tr>
</tbody>
</table>