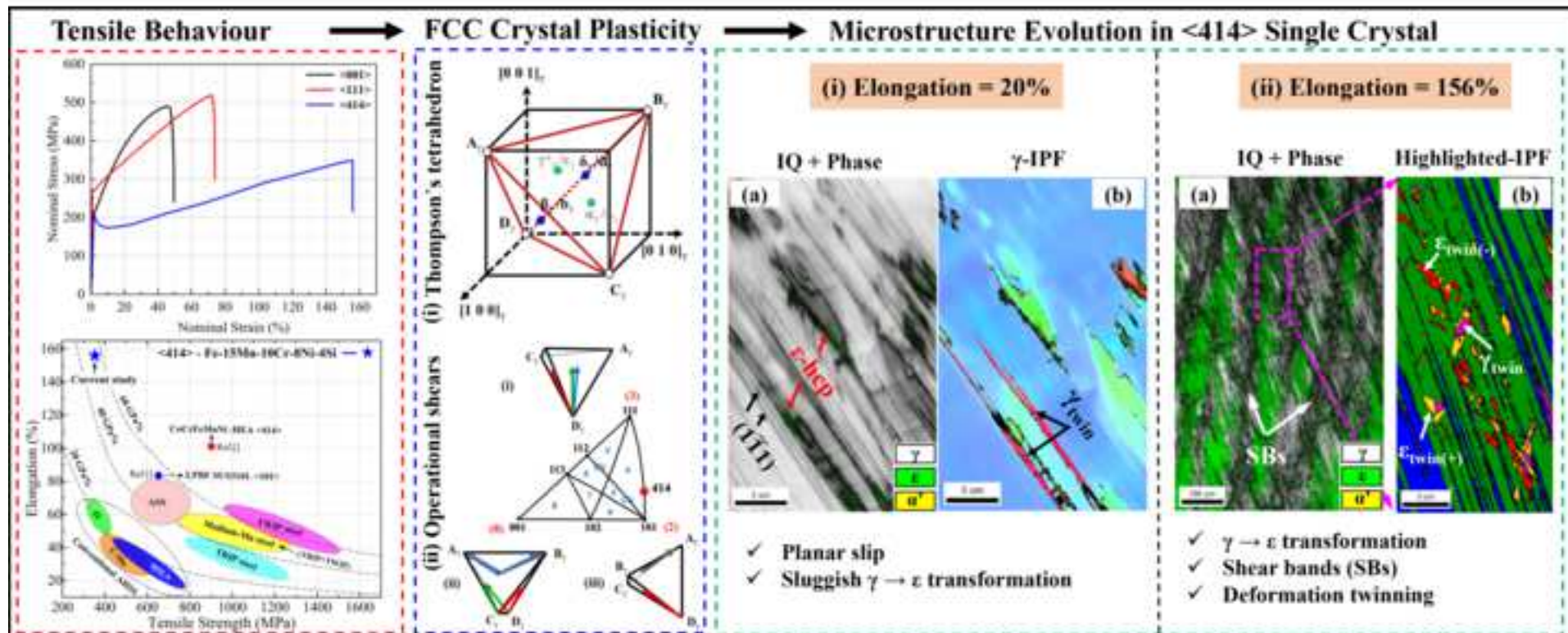




Revealing Extraordinary Plasticity in Single Crystal Fe-15Mn-10Cr-8Ni-4Si Alloy

Digvijay Singh¹, Fumiyoshi Yoshinaka¹, Susumu Takamori¹, Satoshi Emura¹, Takahiro Sawaguchi^{1*}

¹ National Institute for Materials Science, 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047, Japan

Digvijay Singh

singh.digvijay@nims.go.jp

Fumiyoshi Yoshinaka

yoshinaka.fumiyoshi@nims.go.jp

Susumu Takamori

takamori.susumu@nims.go.jp

Satoshi Emura

emura.satoshi@nims.go.jp

Takahiro Sawaguchi*

sawaguchi.takahiro@nims.go.jp

* Corresponding author

Abstract

An extraordinary elongation of over 156 % was achieved in single crystal Fe-15Mn-10Cr-8Ni-4Si alloy, which shows $\gamma \rightarrow \epsilon \rightarrow \alpha'$ transformation. Tensile tests were conducted along the $\langle 414 \rangle$, $\langle 111 \rangle$, and $\langle 100 \rangle$ directions, and the deformed microstructure was characterized with electron-backscattering diffraction (EBSD). Elongation in $\langle 414 \rangle$ orientation (156%) was highest as ever recorded in metallic alloy—approximately twice that of conventional TWIP steels. Mechanical behavior in $\langle 414 \rangle$ is characterized with slow work hardening, with upward curvature in the stress–strain curve. EBSD analysis revealed 45% of ϵ -martensite and significant γ -twin microstructure components in $\langle 414 \rangle$. In initial stage, deformation was marked by planar slip and a sluggish $\gamma \rightarrow \epsilon$ transformation, activating a single ϵ -martensite variant with a Schmid factor of 0.5. Later stages witnessed crystal rotation towards $\langle 112 \rangle$, generating multiple shear bands and distinct ϵ variants. Fractures predominantly occurred along the $\langle 011 \rangle$ direction, with the fracture surface exhibiting a ductile nature.

Keywords: Extraordinary plasticity, Damping steel, Single crystal, Martensitic transformation, Shear band

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3 In recent year, Fe–Mn–Si-based alloys are renowned for their shape memory
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5 effect, owing to their unique $\gamma \rightarrow \varepsilon$ martensitic transformation characteristics when it
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7 experiences specific stimuli, such as temperature or stress [1,2]. Recently, a novel Fe–
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9 15Mn–10Cr–8Ni–4Si seismic damping alloy is developed with excellent Low-cycle
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11 (LCF) fatigue properties [3,4]. The fundamental idea behind designing this alloy is to
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13 utilize the reversible $\gamma \leftrightarrow \varepsilon$ transformation under cyclic loading, which was discovered in
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15 high Mn-steels and shape memory alloys [5–18]. Reversible $\gamma \leftrightarrow \varepsilon$ transformation is
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17 considered beneficial because it can retardative crack initiation and propagation, leading
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19 to prolonged LCF life [17]. Recently we identified that the necessary thermodynamic
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21 condition for achieving reversible $\gamma \leftrightarrow \varepsilon$ transformation is $\Delta G^{\gamma \rightarrow \varepsilon} \leq 0$ [4,18].
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27 Fe-15Mn-10Cr-8Ni-4Si alloy represents a class of medium-Mn austenitic steels
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29 with low-stacking-fault-energy ($\Gamma_{\text{SFE}} \sim 19 \text{ mJ/m}^2$) and minimal difference between Gibbs
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31 free energy between γ - and ε -phase ($\Delta G^{\gamma \rightarrow \varepsilon} \sim 12.8 \text{ J/mol}$) [4]. With a low SFE, this alloy
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33 exhibits an impressive strength-ductility balance (51.4 GPa%), as evidenced by a total
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35 elongation of 77% [6]. This performance is comparable to that of twinning-induced
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37 plasticity (TWIP) steels, due to the diverse range of martensitic transformations ($\gamma \rightarrow \varepsilon$,
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39 $\gamma \rightarrow \varepsilon \rightarrow \alpha'$, or $\gamma \leftrightarrow \varepsilon$) and deformation twinning mechanisms [19,20]. During the $\gamma \rightarrow \varepsilon$
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41 martensitic transformation, organized movement of $\{111\} \langle 121 \rangle_{\gamma}$ Shockley partials takes
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43 place within $\{111\}$ close-packed planes.
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50 It is widely acknowledged that the crystallographic orientation can greatly
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52 influence the tensile properties by directly affecting the dominant deformation
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54 mechanisms [21–26]. Wang *et al.* demonstrated orientation-dependent tensile behavior in
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56 316L stainless steel single crystals made by laser powder bed fusion [21]. Dislocation slip
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dominated <001> oriented samples, while twinning dominated <111> samples. UTS and elongation were 645 MPa and 36% for <001>, and 840 MPa and 58% for <111>. Following this, we have investigated the tensile properties of Fe-15Mn-10Cr-8Ni-4Si seismic damping alloy single crystal in different crystallographic directions. The deformation was made in <001>, <111>, and <414> directions. In single crystals, strain hardening behavior is closely associated with the activation of secondary slip systems. The influence of these secondary slips on strain hardening capacities varies significantly depending on the crystal orientation [24,27]. In this paper, we report the extraordinarily high ductility (156%) of novel Fe-Mn-Si damping alloy when deformed in the <414> crystallographic direction. In-depth EBSD microstructural analysis was performed to unveil the mechanisms behind this remarkable ductility.

A single crystal Fe-15Mn-10Cr-8Ni-4Si (mass %) alloy was produced at 1823 K in an inert gas environment using the Bridgman method. Quasi-static tensile tests were conducted in the <001>, <111>, and <414> crystallographic directions at an initial strain rate of $1.4 \times 10^{-3} \text{ s}^{-1}$. The strain was measured by a non-contacting video extensometer (TRView X, Shimadzu Corp.). EBSD microstructure analysis was performed on the deformed specimen's normal planes after surface preparation, which involved electrolytic polishing using perchloric acid solution. A JEOL JSM-7900F field-emission scanning electron microscope was used for EBSD analysis at 20 kV acceleration voltage, with a 15 mm working distance and a step size of 50 nm to 1 μm .

Figure 1 illustrates a detailed orientation-dependent mechanical property of the Fe-15Mn-10Cr-8Ni-4Si alloy. The engineering stress-strain curves for all three different orientations are depicted in Fig. 1(a). Notably, the <414> oriented single crystal exhibits

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2 extraordinary ductility, approximately 156%, which is approximately twice as high as that
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4 of the $\langle 111 \rangle$ crystal (74%) and nearly three times larger (49.8%) than the crystal oriented
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6 along the $\langle 001 \rangle$ direction. The maximum ultimate tensile strength (UTS) of 520 MPa
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8 and the minimum UTS of 350 MPa are observed for the $\langle 111 \rangle$ and $\langle 414 \rangle$ oriented single
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10 crystals, respectively. Noteworthy, in the $\langle 414 \rangle$ orientation, a significant drop in the yield
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12 stress is observed, which markedly differs from the other directions.
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18 The strain hardening behavior of tensile-deformed single crystals is illustrated in
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20 Fig. 1(b, c). During the initial stage of deformation, the $\langle 001 \rangle$ orientation exhibits a
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22 maximum strain-hardening rate (SHR, $d\sigma/de_T$), while the $\langle 414 \rangle$ direction demonstrates
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24 the minimum. In the $\langle 001 \rangle$ orientation, the SHR quickly peaks, leading to a shorter initial
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26 stage. Around true strain, $e_T \approx 0.08$, it transitions directly to stage 3 hardening, where the
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28 rate gradually decreases until necking at $e_T \approx 0.37$. The SHR vs. true strain (e_T) curve for
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30 the $\langle 414 \rangle$ crystal is shown in Fig. 1(c). Initially, the curve indicates easy glide up to $e_T \approx$
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32 0.13 (region-II). This is followed by a significant secondary hardening stage (region III),
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34 with the SHR gradually increasing to a maximum at $e_T \approx 0.92$. In the $\langle 111 \rangle$ orientation,
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36 strain hardening also shows three distinct regions similar to the $\langle 414 \rangle$ orientation.
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38 However, region III has higher strain hardening, extending to a shorter true strain of about
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40 0.52. Furthermore, the Considère criterion for plastic instability ($d\sigma/de_T \leq \sigma$) is met only
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42 for crystals oriented along the $\langle 001 \rangle$ direction. Figure 1(d) provides a comprehensive
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44 comparison of the strength-ductility trade-offs observed across commonly used single and
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46 polycrystalline materials. Remarkably, the $\langle 414 \rangle$ oriented specimen exhibits an
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48 exceptional strength-ductility balance surpassing the 50 GPa% threshold.
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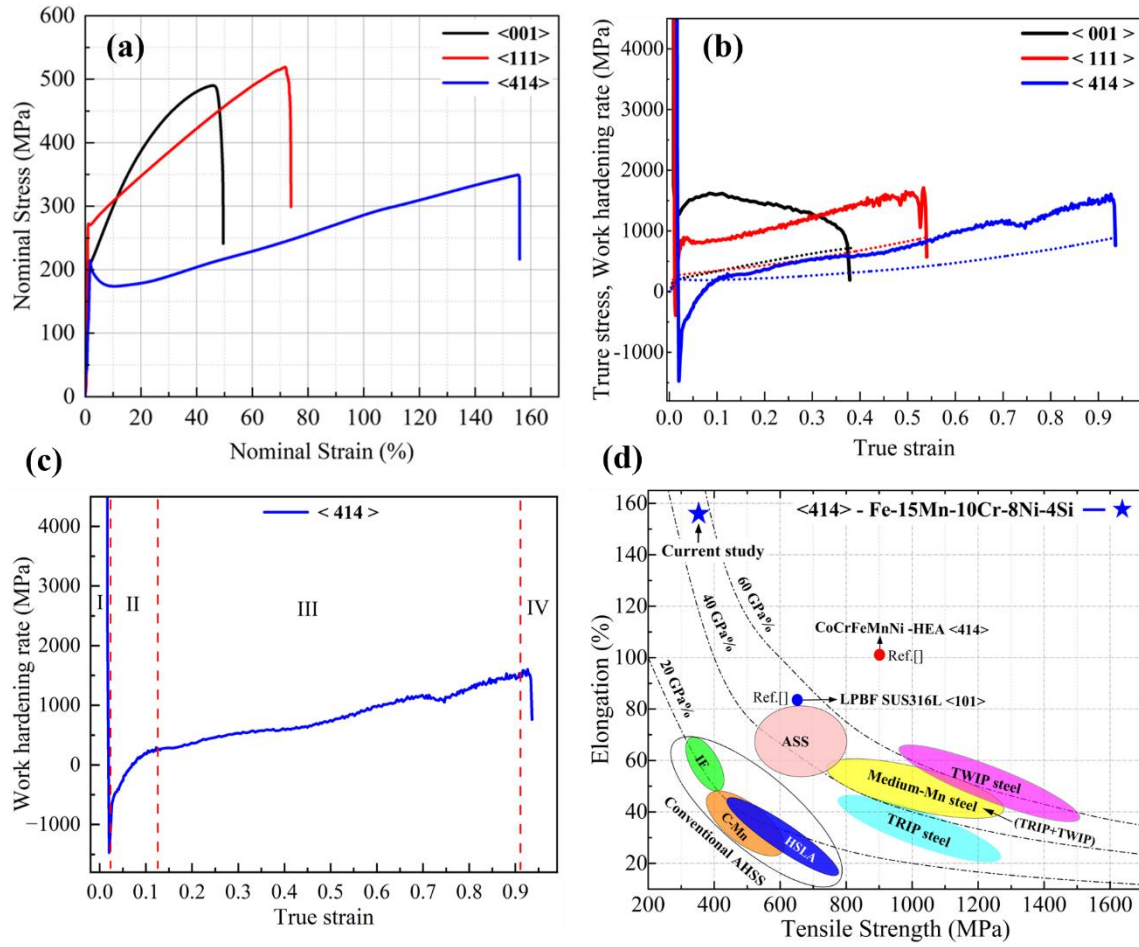


Figure 1. (a) Engineering stress-strain curves, and (b) strain-hardening rate/true stress vs. true strain curves of tensile deformed specimens in the $[414]$, $[111]$, and $[001]$ directions. (c) Work hardening stages in $[414]$ oriented tensile deformed single crystal. (d) Comparison of the strength-ductility trade-off reported in various steels and the current study.

Figure 2 presents a detailed EBSD microstructural analysis of a tensile-fractured single crystal oriented in various directions. It includes a colored phase map for three different orientations, depicted in Fig. 2(a), 2(c), and 2(e), and their respective inverse pole figure (IPF) maps in Figure 2(b), 2(d), and 2(f). Following tensile deformation, martensitic transformation occurs in all crystallographic directions, albeit with a highly orientation-dependent nature. The orientation-dependent martensitic transformation can be predicted using Schmid's criterion with Thompson's tetrahedron, as shown in Fig. 2(g).

Figure 2(h) shows all potential shear systems for martensitic transformation within the $[001]$ – $[101]$ – $[111]$ standard triangle, indicated by arrows pointing towards or away from the gravity center relative to the tensile loading direction.

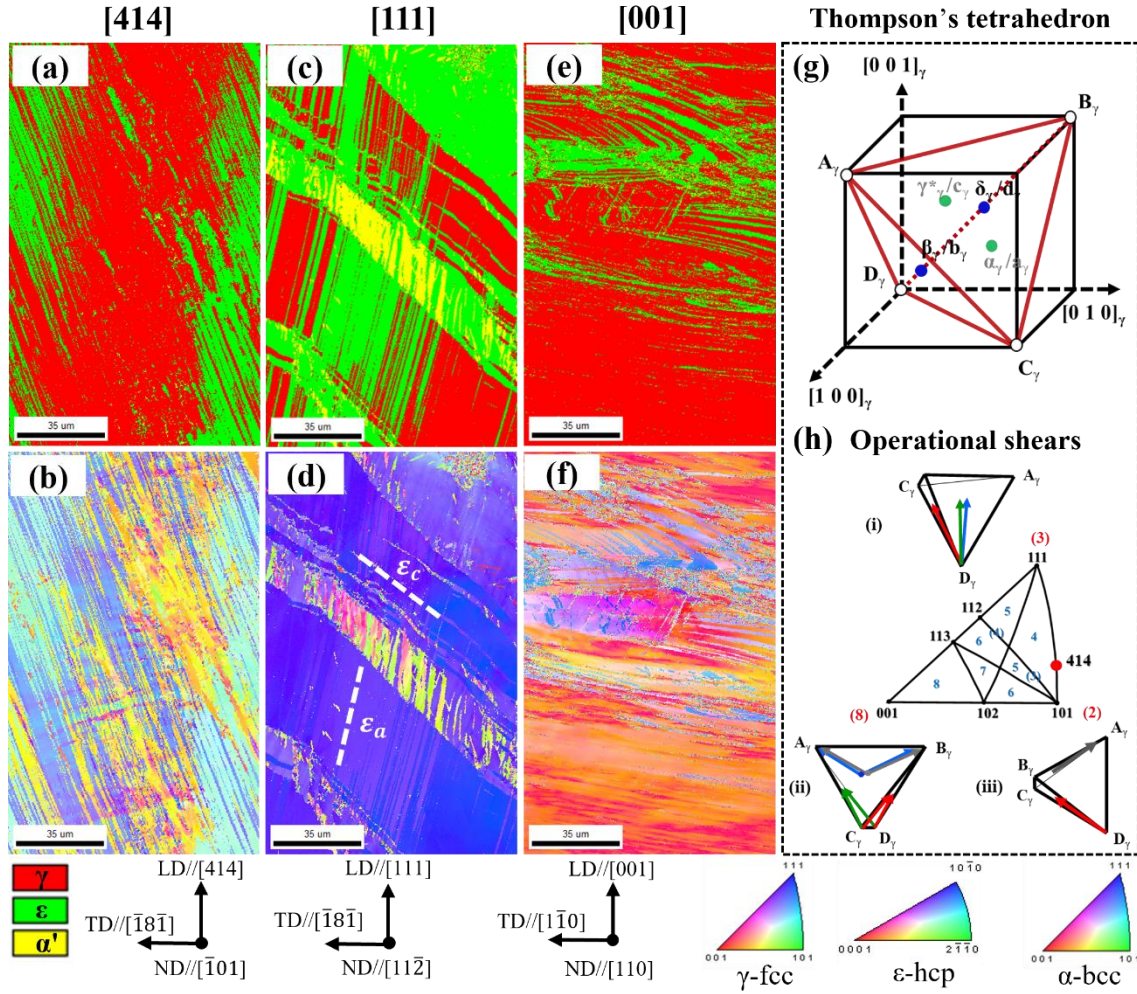


Figure 2. (a, c & e) EBSD phase map and (b, d & f) corresponding inverse pole figure (IPF) map of $[414]$, $[111]$, and $[001]$ oriented tensile fractured single crystals, respectively. (g) Illustration of regular Thompson's tetrahedron. (h) Plausible operational shear systems (Shockley partials) in the standard $[001]$ – $[101]$ – $[111]$ triangle under tensile deformation.

Table 1 displays the Schmid factors for the operative martensitic shears in each of the three crystal directions. In the $\langle 414 \rangle$ orientation, two primary partials demonstrate

notably high Schmid factors of 0.5 ($D\alpha/a$) and 0.39 ($\delta A/\bar{d}$). Similarly, the three shears in $\langle 111 \rangle$ and eight shears in $\langle 001 \rangle$ orientation display equal Schmid factors of 0.31 and 0.24, respectively.

Table 1. *Plausible $\gamma \rightarrow \varepsilon$ shear systems (partial dislocations) in various crystallographic orientations and their corresponding Schmid factor values.*

Orientation	Operational shear systems (Schmid Factor)
001	$C\alpha/a$: 0.24, $D\alpha/a$: 0.24, $D\beta/b$: 0.24, $C\beta/b$: 0.24, $\gamma A/\bar{c}$: 0.24, $\gamma B/\bar{c}$: 0.24, $\delta A/\bar{d}$: 0.24, $\delta B/\bar{d}$: 0.24
111	$D\alpha/a$: 0.31, $D\beta/b$: 0.31, $D\gamma/c$: 0.31
414	$D\alpha/a$: 0.50, $\delta A/\bar{d}$: 0.39

After tensile fracture, approximately 46% single-variant ε -martensite is observed in the $\langle 414 \rangle$ oriented specimen (Fig. 2(a, b)). In the $\langle 111 \rangle$ tensile-deformed microstructure, a similar volume fraction of ε -martensite ($47 \pm 3\%$), consisting of two variants (ε_a , ε_c), is observed (Fig. 2(c, d)). Unlike $\langle 414 \rangle$, Unlike $\langle 414 \rangle$, the $\langle 111 \rangle$ direction also shows an additional α' -bcc martensite phase. It has been established that α -bcc martensite is observed at specific sites where two different ε -martensite variants intersect each other [20,28]. The IPF map in Fig. 2(d) reveals the formation of multiple Kurdjumov–Sachs (K–S) variants at the ε - ε martensite intersection: V22 ($(1 \ 1 \ \bar{1})_\gamma // (0 \ 1 \ 1)_{\alpha'}$, $[1 \ 0 \ 1]_\gamma // [\bar{1} \ \bar{1} \ 1]_{\alpha'}$) and V23 ($(1 \ 1 \ \bar{1})_\gamma // (0 \ 1 \ 1)_{\alpha'}$, $[1 \ 0 \ 1]_\gamma // [\bar{1} \ \bar{1} \ 1]_{\alpha'}$). The

determination of K–S variants at these intersection sites is discussed in detail in our previous paper [28]. In the $\langle 001 \rangle$ orientation, about 21% ϵ -martensite is observed in the deformed microstructure, with no α' -bcc phase present.

To investigate the microstructural evolution and deformation mechanism of the $\langle 414 \rangle$ single crystal, tensile tests were halted at 20%, 100%, and 156% (fractured strain). Figure 3 presents the superimposed phase map with the image quality (IQ) map, IPF map, and kernel average misorientation (KAM) maps for these strain levels. A lamellar banded structure comprising a single ϵ -martensite variant (green) aligned in the primary operative shear ($D\alpha/a$) direction is observed along the parent γ -austenite phase (white). At 20% strain, only $3 \pm 1\%$ ϵ -martensite is detected in the microstructure (Fig. 3(a)). The IPF map (Fig. 3(b)) shows a single color with a slight gradient, revealing minimal angular rotation of the γ -phase. At 100% strain, ϵ -martensite reaches its maximum volume of about $45 \pm 5\%$. The deformation twins in the microstructure increases with higher strain levels.

In addition to uniform deformation, significant localized shear band (SB) formation occurs in the $\langle 414 \rangle$ crystal during later stages of deformation. These SBs intensify with increased tensile straining, as depicted in Fig. 3 (d-i). SBs generate severe strain fields, resulting in heightened misorientation and crystal rotation, as demonstrated in the KAM map (Fig. 3(f, i)). High magnification phase and IPF maps of a highlighted region (Fig. 3(j-k)) show very fine $\Sigma 3$ -type γ -twins (pink) at the γ/ϵ interface, and $\{10\bar{1}2\}$ type fine ϵ -twins growing in strained ϵ -martensite region. The IPF map (Fig. 3(k)) reveals two distinct colors (yellow and red) representing the 86° (+ve twin) and 94° (-ve twin) ORs with the original ϵ -martensite phase. Corresponding pole figures (Fig. 3(l-m))

establish the Shoji–Nishiyama crystallographic orientation relationship among the γ - ϵ phases: $\{1\ 1\ 1\}_{\gamma} // \{0\ 0\ 0\ 1\}_{\epsilon}$, $\langle 1\ 0\ \bar{1} \rangle_{\gamma} // \langle 2\ \bar{1}\ \bar{1}\ 0 \rangle_{\epsilon}$.

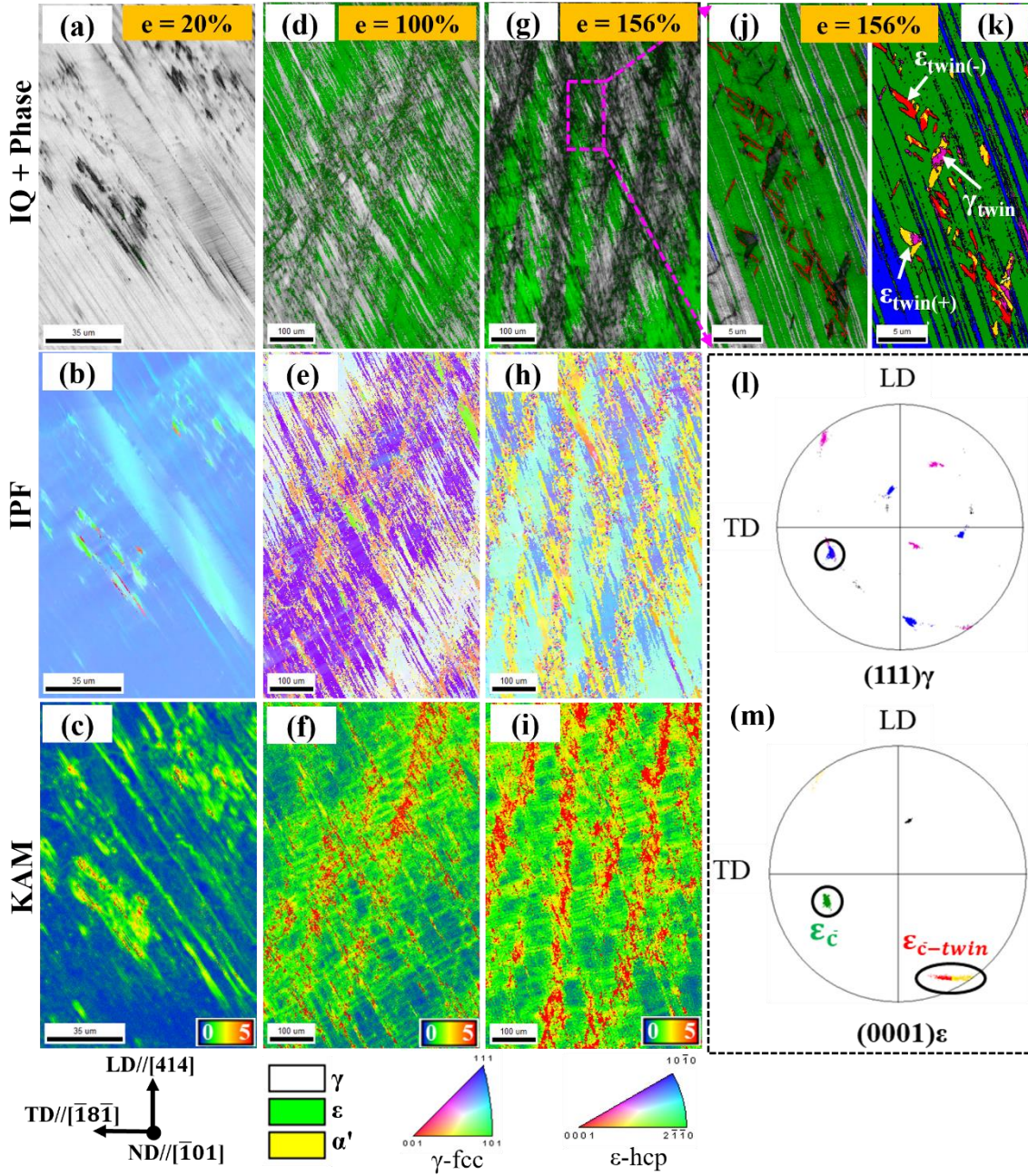


Figure 3. a, d, g) IQ + phase maps, (b, e, h) IPF maps, and (c, f, i) kernel average misorientation (KAM) maps of [414] single crystal specimens with tensile strains of 20%, 100%, and fractured strain (156%), respectively. (j) High magnification IQ + phase map,

(k) highlighted IPF map, and (l-m) corresponding pole figure maps showing the orientation relationship between γ -austenite (γ), γ -twin, and ε -martensite variant c (ε_c).

In addition to uniform deformation, significant localized shear band (SB) formation occurs in the $\langle 414 \rangle$ crystal during later stages of deformation. These SBs intensify with increased tensile straining, as depicted in Fig. 3 (d-i). SBs generate severe strain fields, resulting in heightened misorientation and crystal rotation, as demonstrated in the KAM map (Fig. 3(f, i)). High magnification phase and IPF maps of a highlighted region (Fig. 3(j-k)) show very fine $\Sigma 3$ -type γ -twins (pink) at the γ/ε interface, and $\{10\bar{1}2\}$ type fine ε -twins growing in strained ε -martensite region. The IPF map (Fig. 3(k)) reveals two distinct colors (yellow and red) representing the 86° (+ve twin) and 94° (-ve twin) ORs with the original ε -martensite phase. Corresponding pole figures (Fig. 3(l-m)) establish the Shoji–Nishiyama crystallographic orientation relationship among the γ - ε phases: $\{1\ 1\ 1\}_\gamma // \{0\ 0\ 0\ 1\}_\varepsilon$, $\langle 1\ 0\ \bar{1} \rangle_\gamma // \langle 2\ \bar{1}\ \bar{1}\ 0 \rangle_\varepsilon$.

Figure 4 displays the optical and SEM images of the tensile fractured $\langle 414 \rangle$ single crystal specimen. Macroscopically, SBs and primary slip traces along the $\langle 112 \rangle$ direction are visible on the normal direction (ND) plane (Fig. 4(a)). However, the final crack propagation occurred on a different plane along the $\langle 011 \rangle$ direction (Fig. 4(b)). The fracture surface, aligned approximately 52° to the $\langle 414 \rangle$ tensile axis, exhibits ductile fracture with fine and shallow dimples. Fig. 4(c) reveals densely packed dimples, ranging from sub-micron to a few micrometers, and proliferating shear band zones in the central fracture surface.

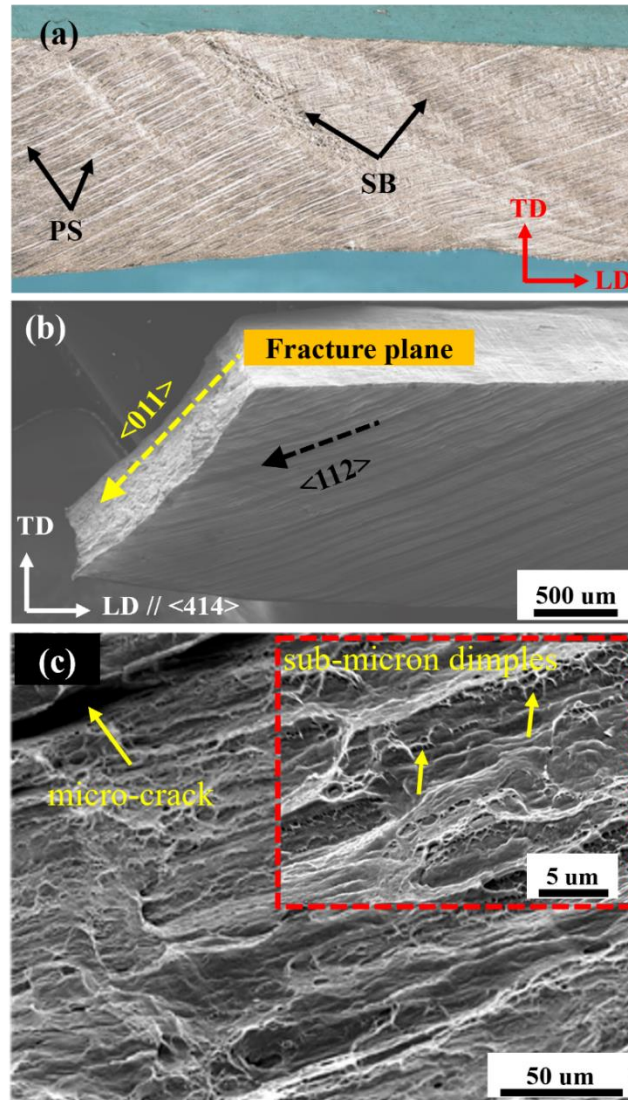


Figure 4. Optical and SEM micrographs of the tensile fractured $\langle 414 \rangle$ oriented single crystal. PS: primary slip band; SB: shear band

The Fe-15Mn-10Cr-8Ni-4Si single crystal oriented in the $\langle 414 \rangle$ direction demonstrated remarkable ductility (156%), nearly doubling that of its polycrystalline alloy counterpart, while maintaining its tensile strength. The yield strength (σ_y) of a single crystal can be estimated using equation (1), which considers the critical resolved shear stress (τ_{CRSS}) and maximum Schmid factor (m) in a specific crystal direction among all operational shear systems [21].

$$\sigma_y = \frac{\tau_{CRSS}}{m} \quad (1)$$

In the Fe-Mn-Si alloy, the Schmid factor is lowest ($m = 0.31$) in $\langle 111 \rangle$ and highest ($m = 0.5$) in the $\langle 414 \rangle$ direction for a given τ_{CRSS} . Therefore, the highest σ_y is expected for the $\langle 111 \rangle$, while the lowest σ_y is expected at $\langle 414 \rangle$ crystal orientation. Multiple slip activation results in the highest strain hardening in the $\langle 001 \rangle$ orientation during the early stages of deformation. Conversely, the $\langle 414 \rangle$ direction experiences easy glide in the initial stage due to primary shears with a maximum Schmid factor ($D\alpha/a = 0.5$), resulting in minimal strain hardening.

The strain hardening response in the $\langle 414 \rangle$ orientation exhibits behavior similar to materials displaying TRIP/TWIP phenomena [22,29]. In the strain hardening curve (Fig. 1(d)), region-I corresponds to dynamic recovery caused by dislocation rearrangement, leading to a sudden drop in the hardening rate at the onset of plastic deformation. Region-II shows a sudden increase in the strain hardening rate, likely due to the activation of planar slip, the primary mechanism of initial stage deformation in the $\langle 414 \rangle$ orientation. The formation of ϵ -martensite and deformation twinning (γ -twin and ϵ -twin) primarily contributed to the large regime hardening observed in region-III in $\langle 414 \rangle$.

To validate the strain hardening mechanism of the $\langle 414 \rangle$ orientation, EBSD analysis was conducted at strain levels of 20% and 156%, as depicted in Fig. 5. At 20% strain, ϵ -martensite appears alongside the less rotated γ region, highlighted in green in the γ -IPF map (Fig. 5b). Additionally, to accommodate plastic strain, γ -twins form along the γ/ϵ interface, shown in red (Fig. 5b). The crystal rotation from $\langle 414 \rangle$ to $\langle 011 \rangle$, depicted in the IPF triangle (Fig. 5e), suggests that martensitic transformation relaxes the rotation

through slip mechanisms. Despite the discrete presence of ε -martensite, continuous slip traces are evident in the (IQ + phase) map (Fig. 5a). Therefore, in the initial stages of deformation, plasticity is primarily driven by $\langle 011 \rangle$ ($\bar{1}\bar{1}1$) planar slip.

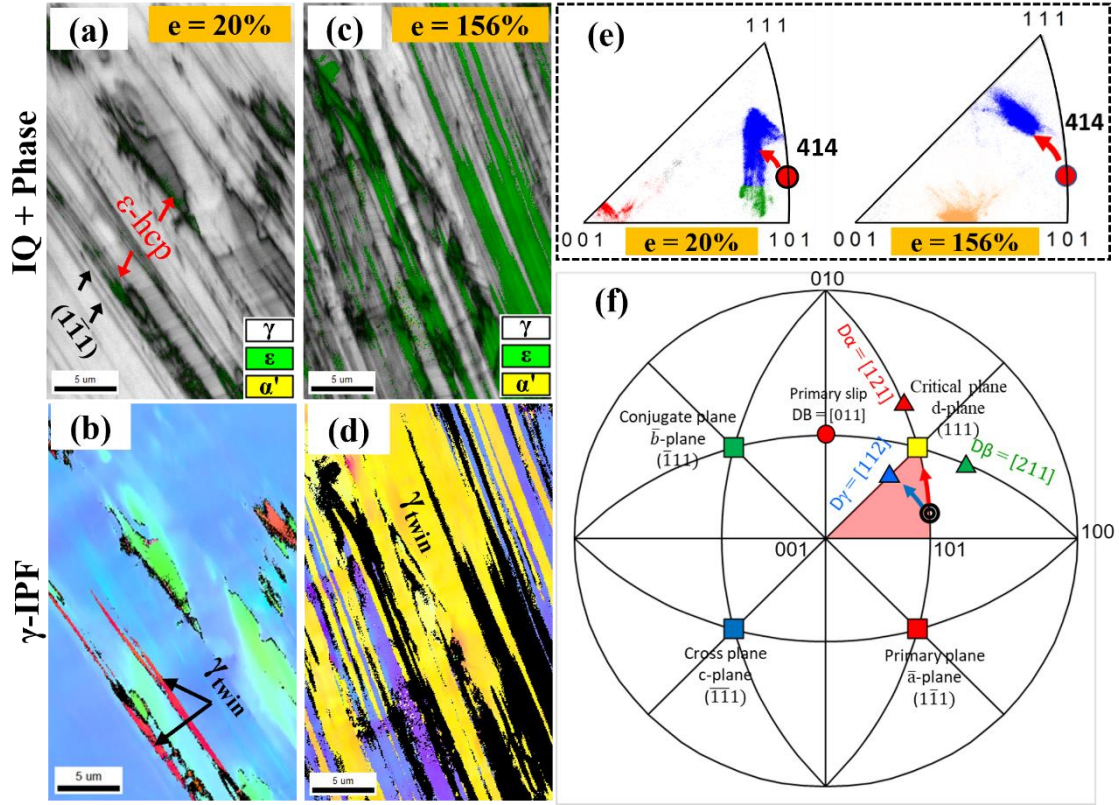


Figure 5. Deformation axis rotation of $[414]$ single crystal at the tensile strain of 20%, and fractured strain (156%). (f) Unique definition of the deformation axis in $[001]$ - $[101]$ - $[111]$.

In the TF specimen, the $\langle 414 \rangle$ microstructure features a significant amount of ε -martensite and γ -twinning. As strain increases, the $\langle 414 \rangle$ tensile axis rotates towards $\langle 112 \rangle$ on the $[001]$ - $[111]$ boundary (Fig. 5e). Similar lattice rotation has been observed in several FCC materials under tensile loading [30–34]. Upon reaching the $[001]$ - $[111]$ boundary, $\langle 101 \rangle$ ($\bar{1}\bar{1}1$) conjugate slip with a high Schmid factor, referred to as secondary slip, is activated along with the primary slip. Fig. 5(f) provides a detailed representation

of the multiple slip systems (primary and conjugates) and operative martensitic shears using the (001) pole figure. Suzuki *et al.* reported that the conjugate system with a lower Schmid factor can activate even if the crystal rotation keeps the tensile axis within the [001]–[101]–[111] standard triangle [30]. Due to the activation of secondary slip systems, localized SBs readily form in materials under high strain conditions [35]. These SBs result from a concentrated accumulation of dislocations, driven by non-uniform plastic deformation, which intensifies strain in specific regions. In addition to TRIP/TWIP phenomena, SBs causes lattice rotation towards the tensile axis, significantly impacting strain hardening [36,37]. In fcc single crystals, SBs drive the activation of conjugate slip systems during stage-II hardening [36]. Crystals with stable orientations are less prone to SB formation during deformation [37]. In <414> crystals, SBs enhanced strain hardening capacity by accommodating significant plastic strain. As dislocations move within SBs, they hinder dislocation motion, leading to strain hardening. Similarly, kink-bands were found to act as superior barriers to dislocation motion compared to grain boundaries, reducing localized failure at low strain levels and extending ductility in Mg-Zn alloys [38,39]. As strain increases, multiple shear bands interact, thereby amplifying strain hardening through the dynamic Hall-Petch effect. Additionally, the SBs in <414> enhanced ductility by providing channels for stress redistribution, thus delaying failure.

In summary, we discovered orientation-dependent exceptional ductility in a Fe-15Mn-10Cr-8Ni-4Si alloy single crystal, reaching approximately 156% in the <414> orientation. The strain hardening behavior in this orientation was characterized by planar slip and a sluggish $\gamma \rightarrow \epsilon$ transformation during the initial stage, followed by the activation of multiple slip systems (secondary/conjugate slip) and shear bands due to crystal rotation in the later stage of deformation. The interaction of nanoscale twinning and shear bands

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2
3 resulted in extremely fine dimples, which contributed to slow crack propagation and
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5 delayed necking. Thus, the remarkable ductility demonstrated by the $\langle 414 \rangle$ orientation
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7 is attributed to the combined effects of planar slip, slow martensitic transformation
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9 kinetics, and the formation of ultra-fine hierarchical structures (twins and SBs). These
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11 factors synergize to prolong strain hardening and contribute to the exceptional ductility
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13 observed in the material. These findings open new possibilities for fabricating ultra-high
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15 ductility alloys.
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30 specimens.
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Declaration of interests

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

☐The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: