



A novel atomic mechanism of fcc $\rightarrow$ hcp $\rightarrow$ bcc phase transition in a gradient nanostructured compositionally complex alloy

Wenqing Yang, Lei Qian, Jiasi Luo & Xu-Sheng Yang

To cite this article: Wenqing Yang, Lei Qian, Jiasi Luo & Xu-Sheng Yang (2024) A novel atomic mechanism of fcc $\rightarrow$ hcp $\rightarrow$ bcc phase transition in a gradient nanostructured compositionally complex alloy, Materials Research Letters, 12:12, 929-938, DOI: [10.1080/21663831.2024.2405228](https://doi.org/10.1080/21663831.2024.2405228)

To link to this article: <https://doi.org/10.1080/21663831.2024.2405228>



© 2024 The Hong Kong Polytechnic University. Published by Informa UK Limited, trading as Taylor & Francis Group.



Published online: 19 Sep 2024.



Submit your article to this journal [↗](#)



Article views: 3925



View related articles [↗](#)



View Crossmark data [↗](#)



Citing articles: 13 View citing articles [↗](#)



ORIGINAL REPORTS



A novel atomic mechanism of fcc $\rightarrow$ hcp $\rightarrow$ bcc phase transition in a gradient nanostructured compositionally complex alloy

Wenqing Yang^{a,b}, Lei Qian^{a,b}, Jiasi Luo^a and Xu-Sheng Yang^{a,b}

^aState Key Laboratory of Ultra-precision Machining Technology, Department of Industrial and Systems Engineering, The Hong Kong Polytechnic University, Kowloon, Hong Kong, People's Republic of China; ^bHong Kong Polytechnic University Shenzhen Research Institute, Shenzhen, People's Republic of China

ABSTRACT

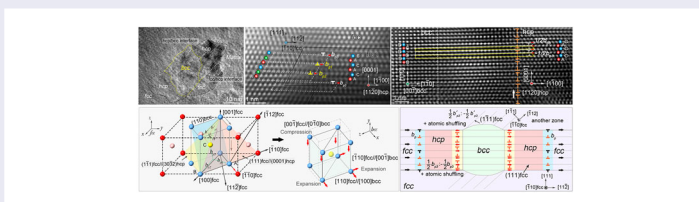
This study investigates the plastic deformation-induced fcc $\rightarrow$ hcp $\rightarrow$ bcc phase transition within nanograins in an ultra-strong gradient nanostructured surface layer on Fe₄₅Mn₃₅Cr₁₀Co₁₀ compositionally complex alloy (CCA), subjected to a single point cubic boron nitride turning process. High-resolution transmission electron microscope observations reveal a novel atomic movement mechanism of this transition following a unique Nishiyama-Wassermann orientation relationship. Unlike the previously validated Bogers-Burgers-Olson-Cohen model, which follows different orientation relationships and relies on two shear components, this study demonstrates that the phase transition in this CCA is cooperatively completed by two sets of half-partial dislocation dipoles and associated atom shuffling.

ARTICLE HISTORY

Received 13 July 2024

KEYWORDS

Compositionally complex alloys; martensitic phase transformation; high-resolution transmission electron microscopy; partial dislocation dipole; atom shuffling



STATEMENT OF NOVELTY

A novel atomic mechanism of fcc $\rightarrow$ hcp $\rightarrow$ bcc phase transition, cooperatively completed by two sets of partial dislocation dipoles and atomic shuffling, provides potential implications for advanced structural materials.

1. Introduction

Solid-to-solid martensitic phase transition, involving collective atomic displacements smaller than the nearest interatomic spacing, plays a crucial role in the mechanical behaviors of materials [1]. When coupled with plasticity, it is referred to as plastic deformation-induced martensitic transformation (PDIMT) [2]. PDIMT, in conjunction with the transformation-induced plasticity (TRIP) effect, significantly contributes to the overall plasticity and work-hardening capabilities of materials [3,4]. The characteristics of PDIMTs, including types, pathways, and underlying atomic movement mechanisms, are highly sensitive to various factors such as composition, microstructure, and process conditions [5]. Based

on Bain correspondence, various hard-sphere shear models have been proposed to describe the atomic movement mechanisms of PDIMTs in metals [6,7]. Our previous studies on gradient nanostructured (GNS) steels have captured atomic-scale evidence of transition features, including lattice rotation, transition lattices, dislocation dipoles, and atom shuffling [8–10]. These results elucidated the atomic mechanisms of polymorphic PDIMTs, such as fcc $\rightarrow$ hcp $\rightarrow$ bcc [11], fcc $\rightarrow$ hcp $\rightarrow$ bct [8,12], and fcc $\rightarrow$ hcp $\rightarrow$ bct $\rightarrow$ bcc [13,14], for the first time validating the 60-year-old Bogers-Burgers-Olson-Cohen (BBOC) model [15] and further developing novel models incorporating partial dislocation dipoles and atom shuffling [16].

CONTACT Xu-Sheng Yang ✉ xsyang@polyu.edu.hk 🏢 State Key Laboratory of Ultra-precision Machining Technology, Department of Industrial and Systems Engineering, The Hong Kong Polytechnic University, Hung Hom, Kowloon, Hong Kong, People's Republic of China; 🏢 Hong Kong Polytechnic University Shenzhen Research Institute, Shenzhen 518060, People's Republic of China

Compositionally complex alloys (CCAs), including high-entropy alloys, represent a new class of advanced structural metals with superior mechanical properties [17–19]. Like the traditional alloys, PDIMTs have been realized to create dual- or multi-phase hetero-structured CCAs, enabling a desirable strength-ductility combination [20–22]. For instance, various PDIMTs, such as fcc $\rightarrow$ hcp in FeMnCrCoNi [21,23], bcc $\rightarrow$ hcp in TiZrHf-TaNb [10,22], fcc $\rightarrow$ bcc in CoNiFeAlCu and FeMn-CrCoNi [24,25], hcp $\rightarrow$ bcc following BBOC model in Fe_{38.5}Mn₂₀Cr₁₅Co₂₀Si₅Cu_{1.5} CCAs [26–30], have been documented to enhance the strength-ductility combination. Notably, the distinct characteristics of CCAs (e.g. compositions) may exert a more complex influence on phase transition characteristics, such as the phase stability and boundaries, transition conditions, pathways and kinetics. Consequently, it can lead to the manifestation of diverse underlying atomic movement mechanisms governing their PDIMTs. As a result, detailed atomic-scale observations are imperative to capture PDIMTs in CCAs and reveal the associated mechanisms.

The present work unveils a notable fcc $\rightarrow$ hcp $\rightarrow$ bcc PDIMT within a GNS surface layer on a Fe₄₅Mn₃₅Cr₁₀Co₁₀ CCA processed by a severe plastic deformation technique, i.e. single point cubic boron nitride turning (SPCBNT) [31]. The ultra-precision SPCBNT process enables an ultrahigh strain rate (even reaching up to $\sim 10^5$ – 10^6 s⁻¹), thus creating high stress and strain fields that refine and strengthen the sample surface [32,33]. Notably, the fcc $\rightarrow$ hcp $\rightarrow$ bcc PDIMT is discerned mainly through high-resolution transmission electron microscope (HRTEM), exhibiting a distinctive Nishiyama-Wassermann (N-W) orientation relationship (OR) [8], different from conventional observations in metals like steels [9]. Consequently, a novel atomic movement mechanism crucial for accomplishing the fcc $\rightarrow$ hcp $\rightarrow$ bcc PDIMT is unveiled in this work, involving the cooperation of two sets of Shockley partial dislocation dipoles and associated atomic shuffling along two directions.

2. Materials and methods

The Fe₄₅Mn₃₅Cr₁₀Co₁₀ CCA samples were synthesized using vacuum arc-melting, followed by hot-rolling (60% at 900°C) with annealing (900°C, 1 h). The plate-shaped samples stuck on a disk holder (diameter of 150 mm) were subjected to 20-pass SPCBNT on a Moore Nanotech 450UPL machine, as illustrated in previous works [34,35]. The process parameters include a spindle speed of 200 rpm, a turning tool rake angle of 20°, a feed rate of 1 mm/min, and a cutting depth of 10 μ m. Microstructures and phase constituents were analysed

by optical microscopy (OM, Zeiss), X-ray diffraction (XRD), electron backscatter diffraction (EBSD), and HRTEM (JEOL JEM-2100F). Plane-view TEM samples at specific depths were prepared by mechanically polishing from the opposite side of the targeted layer, followed by thinning using the Gatan 691 precision ion polishing system. Mechanical properties at various depths were assessed through nanoindentation and micro-pillar compression tests using the Hysitron TI 900 system. During nanoindentation, a maximum load of 10 mN was applied with a loading/unloading rate of 0.5 mN/s and a holding time of 5 s. For micro-pillar compression tests, pillars with a diameter of 800 nm, a height of 1.8 μ m, and an aspect ratio of 2.5 were prepared using a Focused Ion Beam (FIB) system (Helios Nanolab 600i, FEI) with coarse milling (9.3–2.5 nA) and fine milling (10–40 pA). Subsequently, the micro-pillar compression tests were carried out with an 11 μ m diamond flat-punch tip under displacement control, reaching a total displacement of ~ 580 nm at a constant strain rate of 1×10^{-3} s⁻¹. Each depth underwent mechanical testing were repeated three times to ensure accuracy and reliability.

3. Results and Discussion

The cross-sectional OM image and EBSD images in Figure 1(a) and (c–e) present the microstructures of the Fe₄₅Mn₃₅Cr₁₀Co₁₀ CCA after the SPCBNT process, revealing a coarse-grained structure (average grain size of ~ 30 μ m) in the matrix region and a gradient refined surface layer (~ 60 μ m thickness) with a high residual strain-induced surface distortion. XRD patterns in Figure 1(b) show a progression in the number and intensity of hcp phase peaks from the matrix to the surface of the SPCBNTed CCA, indicating the occurrence of the fcc $\rightarrow$ hcp phase transition during the gradient refinement process and the resultant increase in hcp phase content. The volume fraction of hcp phase gradually increases to $\sim 29\%$, calculated based on integrated intensities of the reflecting planes [36–38]. The nanohardness and compressive stress–strain curves at different depths are shown in Figure 1(f–g), demonstrating an increase in nanohardness from ~ 4.3 GPa in the matrix core to ~ 6.7 GPa at the surface. The stress–strain curves reveal an enhanced yield strength as the depth approaches the surface, with the yield strength at the surface being ~ 2.4 times higher than that in the matrix region. Additionally, the observed reduction in average grain size from ~ 30 μ m in the matrix core to ~ 13 nm at the surface, based on TEM observations. The gradual enhancement of hardness and compressive strength should be attributed to the combination of grain refinement/boundary strengthening and TRIP effects along the depth direction. Several

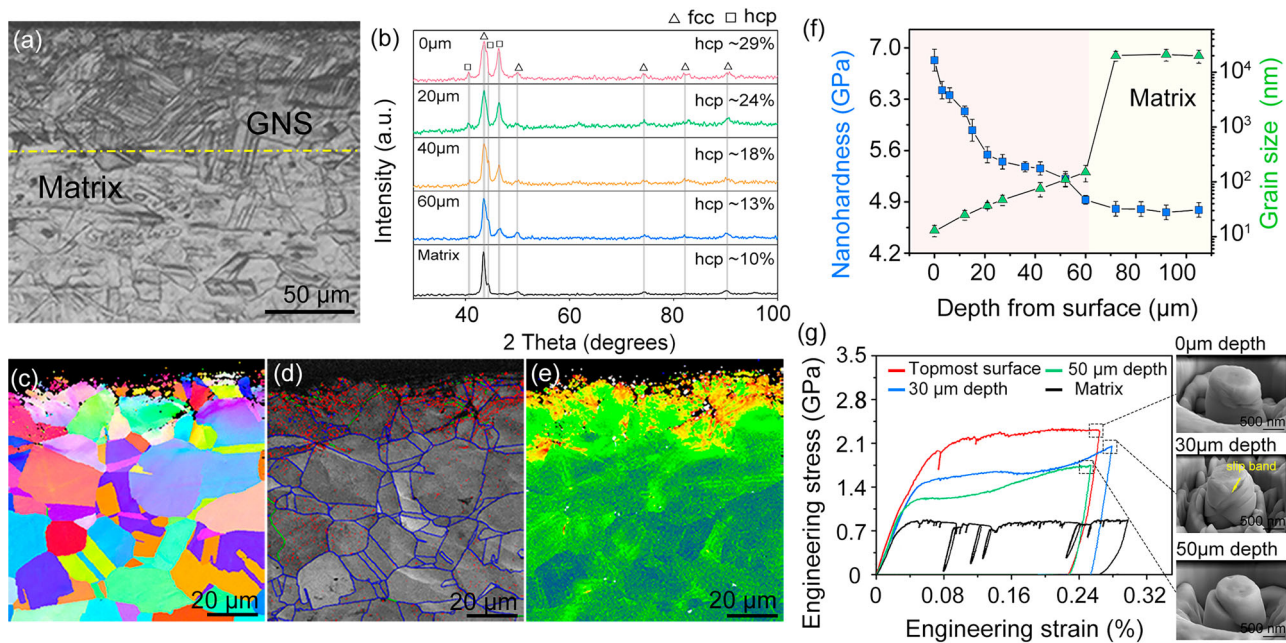


Figure 1. (a–f) Cross-sectional (a) OM image, (b) XRD patterns at different depths. (c) IPF map, (d) grain boundary map, and (e) Kernel average misorientation (KAM) map of the SPCBNTed CCA. (f) The variations in grain size and nanohardness with depths. (g) Micro-pillar compressive engineering stress–strain curves and SEM images of the deformed pillars at different depths.

obvious load drops exhibit in some micro-pillar compressive stress–strain curves, especially at matrix region and topmost surface layer. The load drops in micro-pillar compressive stress–strain curves might be related to the activities of shear bands, dislocation, and phase transition, influenced by some factors such as grain orientation, and ratio between diameter of pillars and grain size [39–41]. When the grain size significantly exceeds the pillar diameter, as in the matrix region, load drops may be easily triggered in the stress–strain curves due to the transient activation of dislocation slip systems. In contrast, the severely refined nanograins (such as $\sim 50 \mu\text{m}$ and $\sim 30 \mu\text{m}$ depth layers) with random orientation would lead to uniform distributions of stress and deformation, potentially alleviating or eliminating the occurrence of load drops. While the recurrence of the slight load drops at the topmost surface with the smallest grain size might be correlated to the relatively large uneven stress distribution through further $\text{fcc} \rightarrow \text{hcp} \rightarrow \text{bcc}$ phase transition activated within some certain fcc-dominated nanograins, which will be observed by HRTEM in the next section. This uneven stress distribution may induce transiently material softening, contributing to the manifestation of load drops. Additionally, SEM images in Figure 1(g) reveal the slip bands in the deformed pillars, indicating the retention of good plasticity in the GNS surface layer.

Representative plane-view TEM images and associated diffraction patterns at different depths along the gradient direction are given in Figure 2 to capture the

phase evolution pathway. Figure 2(a) only detects fcc phase in the core matrix region, while plate-shaped hcp phases are observed at the depths of $\sim 40 \mu\text{m}$ and $\sim 20 \mu\text{m}$, as illustrated in Figure 2(b–c). Moreover, bright- and dark-field TEM images, along with the SAED diffraction pattern in Figure 2(d–e), demonstrate the formation of nanoscale hcp laths within the further refined fcc nanograins at the topmost surface layer. The atomic Fourier-filtered and HRTEM images in Figure 2(f–g) are magnified from regions marked by yellow circles in dark-field TEM images in Figure 2(e). Figure 2(f) reveals the dense stacking faults within these nanoscale hcp laths. Interestingly, Figure 2(g) provides evidence that the nanoscale hcp laths can act as an intermediate phase, serving as nucleation sites for the subsequent formation of the bcc phase within the nanograins. Additionally, the enlarged HRTEM images of the phase interfaces in Figure 2(h–i) and corresponding fast Fourier transform (FFT) diffraction patterns in Figure 2(j–l) confirm the presence of the transformed phases, thus supporting the existence of a polymorphic $\text{fcc} \rightarrow \text{hcp} \rightarrow \text{bcc}$ pathway during the grain refinement process.

Figure 3(a–b) illustrates another typical case of polymorphic $\text{fcc} \rightarrow \text{hcp} \rightarrow \text{bcc}$ phase transition within a nanograined fcc matrix phase. Like the observations in Figure 2(f–g), a smaller nanosized hcp lath is formed with involvement of a series of Shockley partial dislocations within an fcc-dominated nanograin, which will

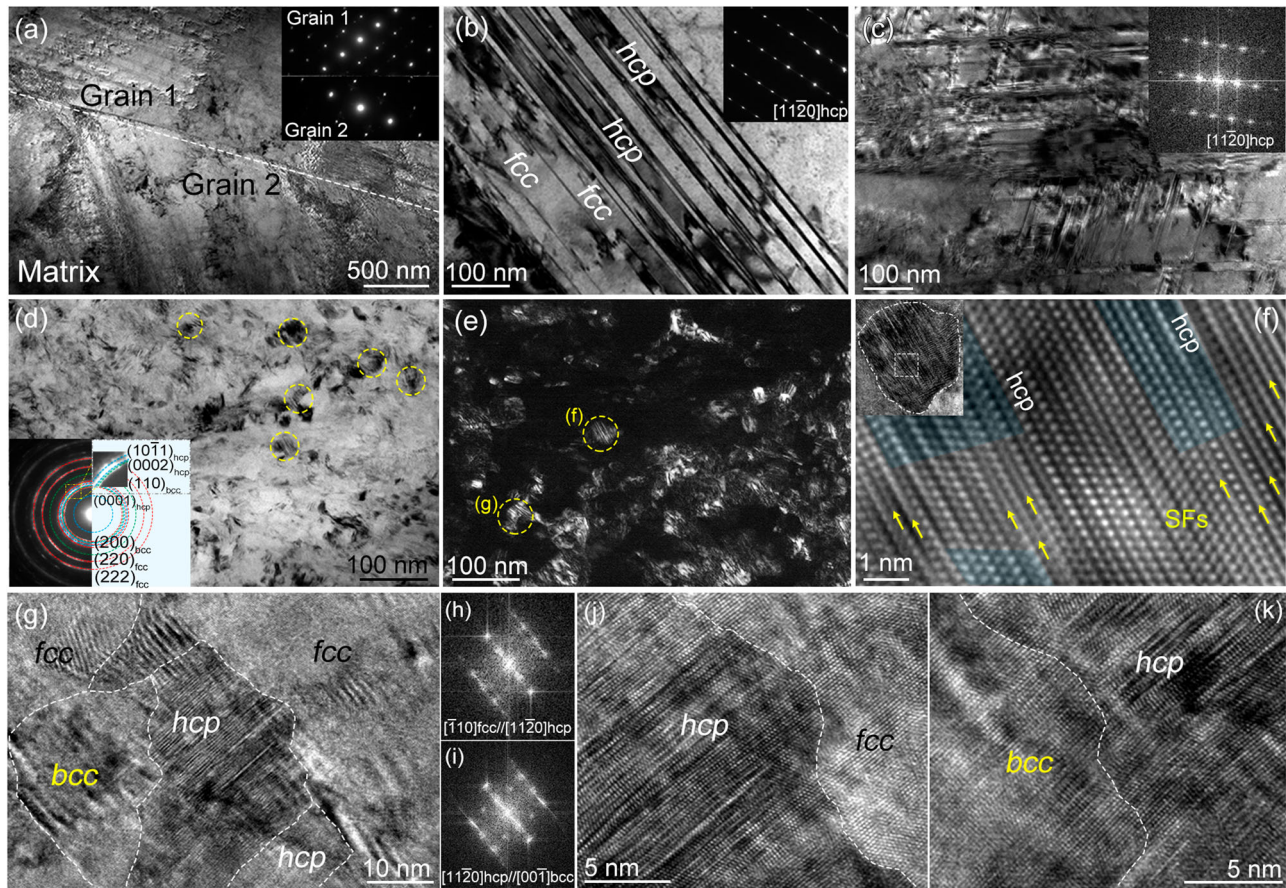


Figure 2. The TEM images with corresponding SAED patterns showing (a) fcc phase in the un-SPCBNTed matrix and (b-c) hcp laths formed at $\sim 40 \mu\text{m}$ and $\sim 20 \mu\text{m}$ depth, respectively. (d) bright-field and (e) dark-field TEM images at the topmost surface layer. (f) An atomic Fourier-filtered image showing the nanoscale hcp laths with dense stacking faults. (g) A typical HRTEM image showing that nanoscale hcp lath is partially transformed to bcc phase. (h-i) FFT diffraction patterns of fcc, hcp and bcc phases and (j-k) Interfaces between fcc phase, nanoscale hcp lath, and bcc phase.

be further atomic-scale dissected in Figure 4. Subsequently, a bcc phase nucleates from this hcp lath, as indicated by the dashed lines in Figure 3(a). Based on the FFT diffraction patterns in Figure 3(b-c), the OR between three phases can be summarized schematically in Figure 3(d). The OR is determined as N-W OR, which follows $(111)\text{fcc}/(0002)\text{hcp}/(110)\text{bcc}$ viewed along $[\bar{1}10]\text{fcc}/[11\bar{2}0]\text{hcp}/[00\bar{1}]\text{bcc}$ directions [8]. The $\text{fcc} \rightarrow \text{hcp} \rightarrow \text{bcc}$ transition in the current SPCBNTed CCA is different from our previous work on stainless steel followed the Pistch OR with $(00\bar{2})\text{fcc}/(\bar{1}01)\text{bcc}$ and K-S OR with $(111)\text{fcc}/(0002)\text{hcp}/(110)\text{bcc}$ under the $[\bar{1}10]\text{fcc}/[11\bar{2}0]\text{hcp}/[1\bar{1}1]\text{bcc}$ directions [9]. The strain distributions within the interfacial regions between the three phases are displayed using a geometric phase analysis (GPA) map in Figure 3(e). It reveals increased out-plane strains (E_{yy}) and significant strain localization in interfacial regions. In addition, Figure 3(f) presents an atomic Fourier-filtered image that directly reveals the atomic structure at the interfacial region between fcc

and bcc, which follows N-W OR [8]. Note that the K-S OR has a inclined angle of $(110)\text{bcc}$ plane to $[\bar{1}12]\text{bcc}$ direction of 60° [9], differing from the inclined angle of $(1\bar{1}0)\text{bcc}$ plane to $[110]\text{bcc}$ direction of 90° for N-W OR, as indicated in Figure 3(f). To determine the bcc lattice constant, an atomic Fourier-filtered image, TEM image and its corresponding FFT pattern of bcc phase projected along the $[1\bar{1}1]\text{bcc}$ direction are provided in Figure 3(g-h). Consequently, Figure 3(i) shows the atomic planar spacing intensity profiles viewed along the $[00\bar{1}]\text{bcc}$ and $[1\bar{1}1]\text{bcc}$ directions, respectively. The lattice constants of three phases are determined as follows: fcc with $a = b = c = \sim 3.81 \text{ \AA}$, hcp with $a = \sim 2.69 \text{ \AA}$, $c = \sim 4.35 \text{ \AA}$, and bcc with $a = b = c = \sim 3.07 \text{ \AA}$, consistent with the reported results [42]. The phase transition characteristics described above indicate an unconventional atomic motion mechanism during the second-step $\text{hcp} \rightarrow \text{bcc}$ transition.

To the best of our knowledge, there are no referenced experiment results reporting the $\text{fcc} \rightarrow \text{hcp} \rightarrow \text{bcc}$

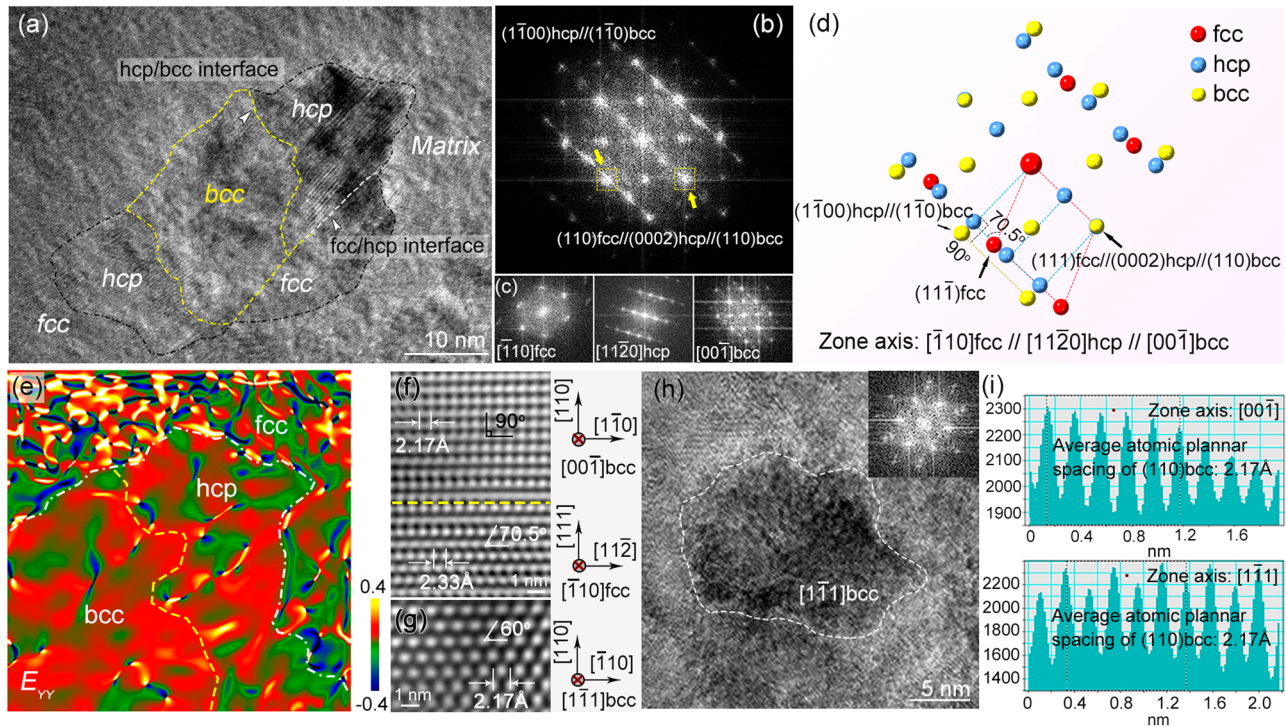


Figure 3. (a) A typical HRTEM image and (b) corresponding FFT diffraction spot showing the fcc $\rightarrow$ hcp $\rightarrow$ bcc PDIMT. (c) FFT diffraction spots for fcc, hcp, and bcc phases along $[\bar{1}10]_{\text{fcc}}$, $[11\bar{2}0]_{\text{hcp}}$, and $[00\bar{1}]_{\text{bcc}}$ directions, respectively. (d) Schematic diagram of the ORs between fcc, hcp, and bcc phases. (e) E_{yy} GPA map analysis of the interfacial region taken from (a). (f) An atomic Fourier-filtered image taken from (a). (g-h) Atomic Fourier-filtered image, TEM image and its corresponding FFT pattern of bcc phase projected along $[1\bar{1}1]_{\text{bcc}}$ direction. (i) Atomic planar spacing intensity profiles of bcc phase projected along $[00\bar{1}]_{\text{bcc}}$ and $[11\bar{1}]_{\text{bcc}}$ directions.

PDIMT in CCAs, particularly with a new OR differing from the previously reported cases, such as in steels [9]. Therefore, it is crucial to uncover the underlying atomic movement mechanisms by dissecting the atomic arrangements and defects present in the interfacial regions. Firstly, Figure 4(a-c) present an HRTEM image and associate FFT patterns, which are enlarged and extracted from Figure 3(a), showing the interface between fcc and hcp phases viewed along the $[\bar{1}10]_{\text{fcc}} // [11\bar{2}0]_{\text{hcp}}$ directions. In Figure 4(d), a further enlarged atomic Fourier-filtered image, extracted from Figure 4(a), reveals that the formation of hcp phase can be completed by the nucleating and gliding of Shockley partial dislocations on every second $(111)_{\text{fcc}}$ planes within the refined fcc-dominated nanograins, as mentioned in Figure 3(a). These partial dislocations have three Burgers vectors (i.e. $\mathbf{b}_{p1} = \frac{a}{6}[2\bar{1}\bar{1}]$, $\mathbf{b}_{p2} = \frac{a}{6}[\bar{1}2\bar{1}]$ and $\mathbf{b}_{p3} = \frac{a}{6}[\bar{1}\bar{1}2]$) on $(111)_{\text{fcc}}$ planes, as indexed in Figure 4(d). They cause different shear components along the $[\bar{1}10]_{\text{fcc}}$ direction but contribute to the same actions to form the hcp lath [15], as schematically shown in Figure 4(e). Secondly, Figure 4(f) and Figure 4(g-h) respectively present an enlarged HRTEM image and associated FFT spots, derived from Figure 3(a), illustrating the hcp $\rightarrow$ bcc transition. An enlarged atomic Fourier-filtered image taken

from Figure 4(f) is provided in Figure 4(i) to examine the atomic arrangements and crystalline defects at the diffuse interface between the hcp and bcc phases. On one hand, it is found that the original inclined angle of $(3\bar{3}02)_{\text{hcp}}$ plane to $[1\bar{1}00]_{\text{hcp}}$ direction, which is $\sim 70.5^\circ$ (a typical group planes following the stacking order of CA//CA//CA...), transforms to an inclination of 90° for $(1\bar{1}0)_{\text{bcc}}$ plane to $[1\bar{1}0]_{\text{bcc}}$ direction, as depicted in Figure 4(j), in accordance with the N-W OR. On the other hand, the atomic spacing along $[11\bar{2}]_{\text{fcc}}$ and $[1\bar{1}00]_{\text{hcp}}$ is equivalent (2.33 Å), which changes to 2.17 Å along $[1\bar{1}0]_{\text{bcc}}$ direction, facilitating the inclined angle change in the fcc $\rightarrow$ hcp $\rightarrow$ bcc transition. Figure 4(k) plots the measured evolution of atomic spacing along the $[1\bar{1}00]_{\text{hcp}}$ direction in the hcp/bcc interfacial region, suggesting that gradual decrease in atomic spacing is completed by the atomic shuffling with localized motions along the $[1\bar{1}00]_{\text{hcp}}$ direction.

Note that gliding any Shockley partial dislocation $\mathbf{b}_p$ ($\mathbf{b}_{p1}$, $\mathbf{b}_{p2}$ or $\mathbf{b}_{p3}$) in the fcc phase can convert the stacking sequence as follows: A $\rightarrow$ B, B $\rightarrow$ C, C $\rightarrow$ A, which completes the fcc $\rightarrow$ hcp transition with... ACACAC... by gliding $\mathbf{b}_p$ on every second $(111)_{\text{fcc}}$ planes. However, gliding $\mathbf{b}_{p1}$ and $\mathbf{b}_{p2}$, unlike $\mathbf{b}_{p3}$, induce shear components along the $[\bar{1}10]_{\text{fcc}}$ direction, causing a

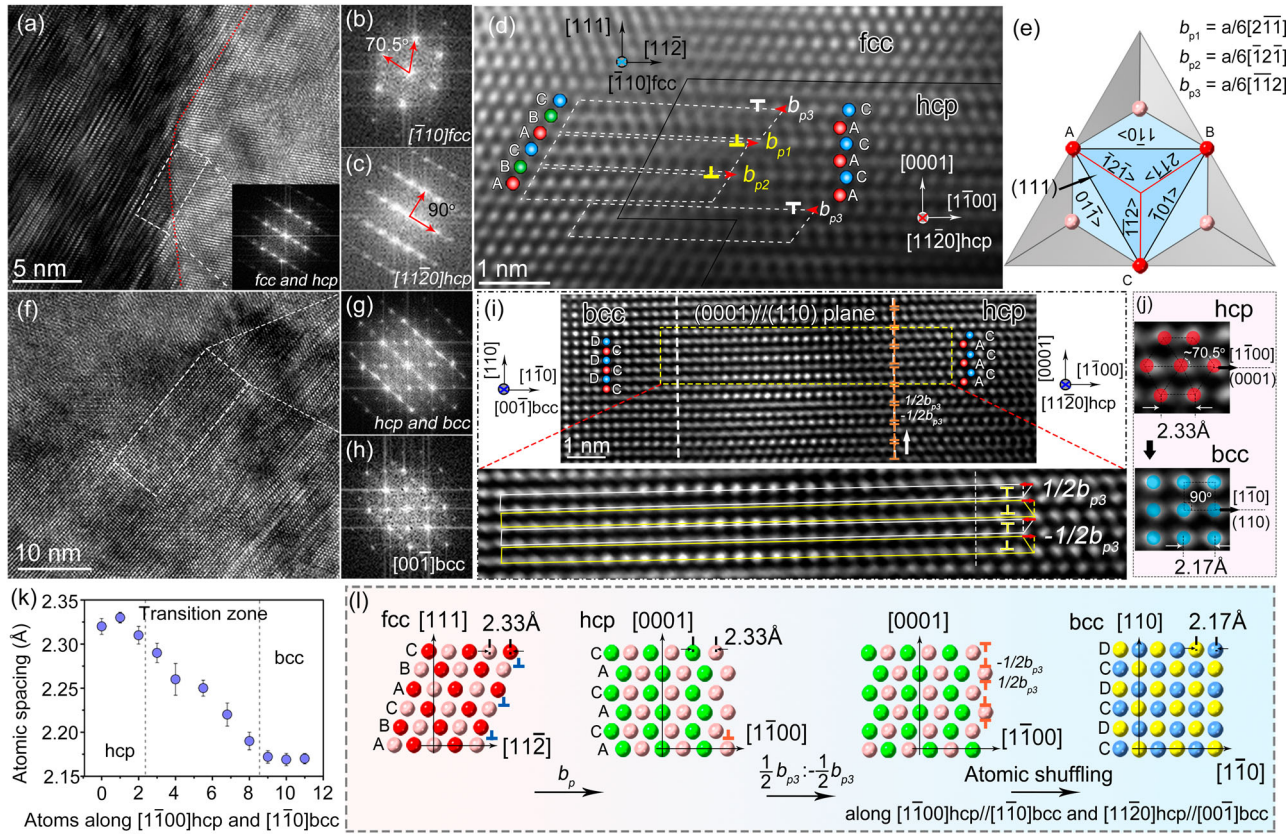


Figure 4. (a) An HRTEM image and the inserted corresponding FFT pattern, and (b–c) corresponding FFT diffraction spots of fcc and hcp phase showing fcc $\rightarrow$ hcp transition. (d) An atomic Fourier-filtered image of the interfacial region between fcc and hcp phases. (e) Schematics showing three types of Shockley partial dislocations on (111)fcc plane. (f) An HRTEM image and (g–h) corresponding FFT spots showing hcp $\rightarrow$ bcc transition. (i–j) Atomic Fourier-filtered images and (k) measured atomic spacing along the $[1\bar{1}00]_{\text{hcp}}$ direction in the diffuse interfacial region between hcp and bcc phases. (l) Schematic illustrating the lattice evolution from fcc to intermediate hcp and finally the bcc phases viewed along $[\bar{1}10]_{\text{fcc}}//[\bar{1}1\bar{2}0]_{\text{hcp}}//[00\bar{1}]_{\text{bcc}}$ directions.

switch in the arrangement of ‘in’ and ‘out’ atoms on $(\bar{1}10)_{\text{fcc}}$ planes. Consequently, only one type of partial dislocation dipoles, namely $-\frac{1}{2}\mathbf{b}_{p3} : \frac{1}{2}\mathbf{b}_{p3}$, can be indexed through the Burgers circuits in Figure 4(i). With $\frac{1}{2}\mathbf{b}_{p3}$ and $-\frac{1}{2}\mathbf{b}_{p3}$ gliding along the two sides of every $(0001)_{\text{hcp}}$ planes in a ‘sandwich’ manner, it can eliminate this switch ‘in’ and ‘out’ status and thus generate the ... CDCDCD ... stacking order in the bcc phase. Figure 4(l) provides a schematic of the lattice evolution from fcc to intermediate hcp and finally the bcc phases viewed along $[\bar{1}10]_{\text{fcc}}//[\bar{1}1\bar{2}0]_{\text{hcp}}//[00\bar{1}]_{\text{bcc}}$ directions. During this process, the inclined angle changes from the original 70.5° for $(11\bar{1})_{\text{fcc}}$ plane to $[11\bar{2}]_{\text{fcc}}$ and eventually reaches 90° for $(1\bar{1}0)_{\text{bcc}}$ plane to $[1\bar{1}0]_{\text{bcc}}$ direction. Through atomic shuffling, the atomic spacing along $[1\bar{1}00]_{\text{hcp}}//[\bar{1}10]_{\text{bcc}}$ directions is gradually reduced from 2.33 Å to 2.17 Å. Additionally, atomic spacing expansion along the $[11\bar{2}0]_{\text{hcp}}//[00\bar{1}]_{\text{bcc}}$ directions is necessary to form the final bcc phase. This atomic movement action will be further illustrated in the following Figure 5 by analyzing the lattice correspondences.

The fcc $\rightarrow$ hcp $\rightarrow$ bcc transition in steels is illustrated by the lattice correspondence in Figure 5(a), which follows the Pistch OR [9] and is completed by the BBOC model the first shear of $\frac{a}{18}[\bar{1}2\bar{1}]$ on every $(11\bar{1})_{\gamma}$ plane and the second shear of $\frac{a}{16}[\bar{1}2\bar{1}]$ on every $(111)_{\gamma}$ plane. They are accommodated by two intermediated hcp phases with slipping dislocations of T/2 on each 111_{γ} plane. The lattice correspondence with the N-W OR [8] is shown in Figure 5(b) for the fcc $\rightarrow$ hcp $\rightarrow$ bcc transition ($a_{\text{bct}} = b_{\text{bct}} \neq c_{\text{bct}}$). One set of partial dislocation dipoles, $-\frac{1}{2}\mathbf{b}_{p3} : \frac{1}{2}\mathbf{b}_{p3}$, on $(111)_{\text{fcc}}$ plane alter the inclined angle and atomic shuffling gradually narrows the atomic spacing along the $[1\bar{1}00]_{\text{hcp}}//[\bar{1}1\bar{2}]_{\text{fcc}}$ directions by 5.88%. It eventually reaches $a_{\text{bct}} = b_{\text{bct}} = \sqrt{2}a_{\text{fcc}}/\sqrt{3}$ while c_{bct} remains unchanged at $c_{\text{bct}} = a_{\text{fcc}}/\sqrt{2}$ throughout the transition process, due to no shear and shuffling actions along the $[\bar{1}10]_{\text{fcc}}//[\bar{1}1\bar{2}0]_{\text{hcp}}//[00\bar{1}]_{\text{bct}}$ directions.

The lattice correspondence for the fcc $\rightarrow$ hcp $\rightarrow$ bcc transition in the current CCA is analyzed in Figure 5(c). The bcc lattice constants are initially $a_{\text{bcc}} = c_{\text{bcc}} = a_{\text{fcc}}/\sqrt{2} = 2.69$ Å and $b_{\text{bcc}} = a_{\text{fcc}} = 3.81$

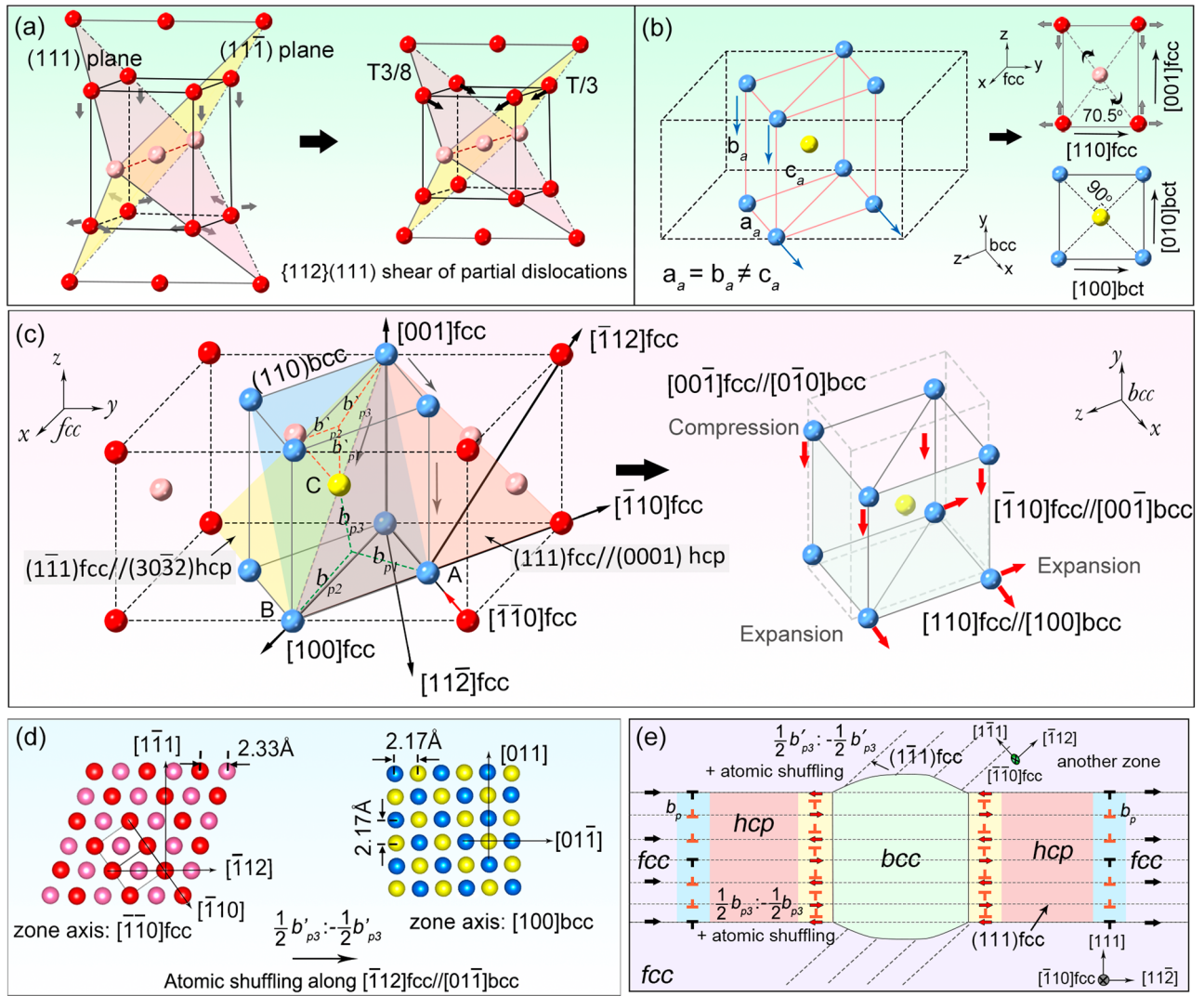


Figure 5. Atomic movement mechanisms schematically illustrated by lattice correspondences. (a) Lattice correspondence for the fcc → hcp → bcc transition completed by the BBOC model [9]. (b) Lattice correspondence for the fcc → hcp → bcc transition completed by one set of partial dislocation dipoles and associated atomic shuffling [8]. (c) Lattice correspondence for the fcc → hcp → bcc transition in the current CCA and (d) lattice evolution projected along $[110]_{fcc}/[110]_{hcp}/[100]_{bcc}$ directions (rotated 90° from the HRTEM view). (e) Schematic diagram of two sets of partial dislocation dipoles associated with atomic shuffling along two vertical directions to form the bcc phase.

Å, and then transits to $a_{bcc} = b_{bcc} = c_{bcc} = 3.07$ Å, following N-W OR as indicated in Figure 3. Obviously, atomic movement mechanism for the current transition differs from the cases illustrated in Figure 5(a) and Figure 5(b), which have either different transition types or different ORs. As observed in Figure 4 and schematically depicted in Figure 5(c), gliding of dislocations is accomplished on two planes: $(111)_{fcc}/(0001)_{hcp}$ and $(1\bar{1}1)_{fcc}/(30\bar{3}2)_{hcp}$ planes. Firstly, the concurrent gliding of dislocation dipoles $-\frac{1}{2}\mathbf{b}_{p3} : \frac{1}{2}\mathbf{b}_{p3}$ on every second $(0001)_{hcp}$ planes and atom shuffling of 6.86% along the $[1\bar{1}00]_{hcp}/[11\bar{2}]_{fcc}$ directions lead to the following change: an increase in the inclined angle of $(1\bar{1}1)_{fcc}$ plane to $[11\bar{2}]_{fcc}$ direction, a compression along

$[00\bar{1}]_{fcc}/[0\bar{1}0]_{bcc}$ directions, and an expansion along $[110]_{fcc}/[100]_{bcc}$ directions. The stacking order and atomic spacing along the $[1\bar{1}0]_{fcc}/[11\bar{2}]_{hcp}/[00\bar{1}]_{bcc}$ directions stay unchanged as these activities exclude shear and shuffling along those directions. However, it is noted that the atomic spacing along $[11\bar{2}]_{fcc}/[1\bar{1}00]_{hcp}/[1\bar{1}0]_{bcc}$ directions should gradually decrease from 2.33 Å to 2.17 Å in Figure 4 (l). Similarly, we propose that concurrent gliding of a second set of partial dislocation dipoles $-\frac{1}{2}\mathbf{b}'_{p3} : \frac{1}{2}\mathbf{b}'_{p3}$ (i.e. $\mathbf{b}'_{p3} = \frac{a}{6}[\bar{1}12](1\bar{1}1)$) on $(1\bar{1}1)_{fcc}$ planes along with atomic shuffling of 6.86% along $[1\bar{1}2]_{fcc}/[01\bar{1}]_{bcc}$ directions, as illustrated in Figure 5(d). The first set of partial dislocation dipoles and related atomic shuffling are perpendicular to these

actions. Subsequently, the second set of partial dislocation dipoles and associated atomic shuffling also induce the change of angle in different planes: an increase in the inclined angle of $(1\bar{1}1)_{\text{fcc}}$ plane to $[\bar{1}12]_{\text{fcc}}$ direction, a compression along $[00\bar{1}]_{\text{fcc}}//[\bar{0}10]_{\text{bcc}}$ directions, and an expansion along $[\bar{1}10]_{\text{fcc}}//[\bar{0}0\bar{1}]_{\text{bcc}}$ directions, respectively. Consequently, $\text{fcc} \rightarrow \text{hcp} \rightarrow \text{bcc}$ transition in the current CCA is cooperatively completed by two sets of partial dislocation dipoles ($-\frac{1}{2}\mathbf{b}_{p3} : \frac{1}{2}\mathbf{b}_{p3}$ on $(111)_{\text{fcc}}$ and $-\frac{1}{2}\mathbf{b}'_{p3} : \frac{1}{2}\mathbf{b}'_{p3}$ and $(1\bar{1}1)_{\text{fcc}}$ planes) and associated atom shuffling of 6.86% (along $[11\bar{2}]_{\text{fcc}}$ and $[\bar{1}12]_{\text{fcc}}$ directions), as schematically shown in Figure 5(e). However, due to the specific transition morphology and perpendicular relation between the two transition actions, it is challenging to observe the second set of dislocation dipoles and associated atomic shuffling using HRTEM, except through atomic movement schematics and simulations [8,43]. Partial dislocation dipoles and atomic shuffling are believed to play crucial role in changing the stacking orders in the low-energy or mobile boundaries, particularly in the low-SFE metals [8,10]. They may also have the ability to locally customize the atomic spacing [10,44]. These transition activities are likely triggered in CCAs with unique and complex characteristics, such as specific compositions, especially under extremely high-loading conditions with ultra-high strain rates [23,45–47]. Based on the Johnson-Cook model [33]: $\sigma = (A + B\varepsilon^n)(1 + C\ln(\frac{\dot{\varepsilon}}{\dot{\varepsilon}_0}))$, the strain ε and strain rate $\dot{\varepsilon}$ generated by the machining process are expressed as: $\varepsilon = \frac{\sqrt{3}}{3}(\frac{\lambda}{\cos\alpha} + \frac{\cos\alpha}{\lambda} - 2 \cdot \tan\alpha)$ and $\dot{\varepsilon} = \frac{d\varepsilon}{dt} = \frac{\varepsilon V}{\Delta}$, respectively [48,49]. In this work, the tool rake angle α is 20° , V is the cutting speed (1570 mm/s), Δ is the deformation zone (60 μm) and $\lambda = \frac{t_c}{t_d}$ (t_c is chip thickness at 8 μm and t_d is cutting depth at 10 μm). The yield strength (A), hardening modulus (B), strain rate sensitivity (C) and hardening coefficient (n) are determined from the dataset as 350, 1344 MPa, 0.013 and 0.312, respectively. The $\dot{\varepsilon}_0$ is the reference strain rate of 1 s^{-1} . Accordingly, the strain rate and equivalent stress of SPCBNT in this work are estimated to be $\sim 4.4 \times 10^4 \text{ s}^{-1}$ and $\sim 2.28 \text{ GPa}$, respectively, which also equivalent to the micro-pillar compressive strength at the top surface ($\sim 2.1 \text{ GPa}$) for the activation of the $\text{fcc} \rightarrow \text{hcp} \rightarrow \text{bcc}$ phase transition. The strain fields induced by high strain rate during machining process could be relieved and accommodated by the $\text{fcc} \rightarrow \text{hcp} \rightarrow \text{bcc}$ PIDMT through the cooperation of dual-directional dislocation dipoles and associated atomic shuffling.

4. Conclusions

In summary, a $\text{fcc} \rightarrow \text{hcp} \rightarrow \text{bcc}$ PIDMT is induced by plastic deformation in an ultra-strong GNS surface layer

on $\text{Fe}_{45}\text{Mn}_{35}\text{Cr}_{10}\text{Co}_{10}$ CCA processed by SPCBNT. This surface layer exhibits exceptional strength enhancement and good ductility, as revealed by micro-pillar compression testing. Based on the HRTEM observations, a novel atomic movement mechanism with a unique Nishiyama-Wassermann orientation relationship is discovered during the $\text{fcc} \rightarrow \text{hcp} \rightarrow \text{bcc}$ transition. Unlike the previously validated BBOC model in steels, the transition in this CCA is accomplished by the cooperation of two sets of half-partial dislocation dipoles and associated atomic shuffling along two vertical directions. These findings provide novel insights into plastic deformation-induced phase transition in CCAs and their potential implications for advanced structural materials.

Disclosure statement

No potential conflict of interest was reported by the author(s).

Funding

This work was supported by the Guangdong Basic and Applied Basic Research Foundation (No. 2022A1515011322), PolyU Grant (No.1-BBRA), Fundamental Research Program of Shenzhen Science and Technology Innovation Commission (No. JCYJ20210324131405015). WY and LQ were supported by the grants from the Research Committee of PolyU under student account codes RK3J and RK2U respectively.

References

- [1] Zhang T, Zhao RD, Wu FF, et al. Transformation-enhanced strength and ductility in a FeCoCrNiMn dual phase high-entropy alloy. *Mater Sci Eng A*. 2020;780: 139182. doi:10.1016/j.msea.2020.139182
- [2] Smaga M, Walther F, Eifler D. Deformation-induced martensitic transformation in metastable austenitic steels. *Mater Sci Eng A*. 2008;483–484:394–397. doi:10.1016/j.msea.2006.09.140
- [3] Li ZM, Tasan CC, Pradeep KG, et al. A TRIP-assisted dual-phase high-entropy alloy: grain size and phase fraction effects on deformation behavior. *Acta Mater*. 2017;131:323–335. doi:10.1016/j.actamat.2017.03.069
- [4] Yang ZM, Yan DS, Lu WJ, et al. A TWIP-TRIP quinary high-entropy alloy: tuning phase stability and microstructure for enhanced mechanical properties. *Mater Sci Eng A*. 2021;801:140441. doi:10.1016/j.msea.2020.140441
- [5] Luo C, Yuan H. Measurement and modeling of deformation-induced martensitic transformation in a metastable austenitic stainless steel under cyclic loadings. *Acta Mater*. 2022;238:118202. doi:10.1016/j.actamat.2022.118202
- [6] Fukui D, Nakada N, Onaka S. Internal residual stress originated from Bain strain and its effect on hardness in Fe–Ni martensite. *Acta Mater*. 2020;196:660–668. doi:10.1016/j.actamat.2020.07.013
- [7] Bowles JS, Wayman CM. The bain strain, lattice correspondences, and deformations related to martensitic transformations. *Metall Trans*. 1972;3:1113–1121. doi:10.1007/BF02642442

- [8] Yang XS, Sun S, Ruan HH, et al. Shear and shuffling accomplishing polymorphic fcc $\gamma \rightarrow$ hcp $\varepsilon \rightarrow$ bct α martensitic phase transformation. *Acta Mater.* 2017;136:347–354. doi:10.1016/j.actamat.2017.07.016
- [9] Yang XS, Sun S, Zhang TY. The mechanism of bcc α' nucleation in single hcp ε laths in the fcc $\gamma \rightarrow$ hcp $\varepsilon \rightarrow$ bcc α' martensitic phase transformation. *Acta Mater.* 2015;95:264–273. doi:10.1016/j.actamat.2015.05.034
- [10] Yang WQ, Luo JS, Fu H, et al. . bcc $\rightarrow$ hcp phase transition significantly enhancing the wear resistance of metastable refractory high-entropy alloy. *Scr Mater.* 2022;221:114966. doi:10.1016/j.scriptamat.2022.114966
- [11] Shahmir H, Asghari-Rad P, Mehranpour MS, et al. Evidence of FCC to HCP and BCC-martensitic transformations in a CoCrFeNiMn high-entropy alloy by severe plastic deformation. *Mater Sci Eng A.* 2021;807:140875. doi:10.1016/j.msea.2021.140875
- [12] He YS, Gao JB, He YZ, et al. The mechanisms of γ (fcc) $\rightarrow \varepsilon$ (hcp) $\rightarrow \alpha'$ (bcc) and direct γ (fcc) $\rightarrow \alpha'$ (bcc) martensitic transformation in a gradient austenitic stainless steel. *J Mater Sci.* 2022;57(8):5230–5240. doi:10.1007/s10853-022-06936-z
- [13] Fu H, Yuan SQ, Sun WT, et al. A novel atomic movement mechanism of intersection-induced bct- $\alpha \rightarrow$ bcc- α' martensitic phase transformation. *Scr Mater.* 2021;204:114153. doi:10.1016/j.scriptamat.2021.114153
- [14] Durandurdu M. Fcc-to-bct phase transformation of aluminum under triaxial stresses: an ab initio constant pressure study. *Eur Phys J B.* 2009;72(2):241–245. doi:10.1140/epjb/e2009-00342-9
- [15] Olson GB, Cohen M. A general mechanism of martensitic nucleation: part II. FCC $\rightarrow$ BCC and other martensitic transformations. *Metall Trans A.* 1976;7:1905–1914.
- [16] Ishii A, Li J, Ogata S. Shuffling-controlled versus strain-controlled deformation twinning: The case for HCP Mg twin nucleation. *Int J Plast.* 2016;82:32–43. doi:10.1016/j.ijplas.2016.01.019
- [17] George EP, Raabe D, Ritchie RO. High-entropy alloys. *Nat Rev Mater.* 2019;4(8):515–534. doi:10.1038/s41578-019-0121-4
- [18] Li WD, Xie D, Li DY, et al. Mechanical behavior of high-entropy alloys. *Prog Mater Sci.* 2021;118:100777. doi:10.1016/j.pmatsci.2021.100777
- [19] Cantor B, Chang ITH, Knight P, et al. Microstructural development in equiatomic multicomponent alloys. *Mater Sci Eng A.* 2004;375:213–218. doi:10.1016/j.msea.2003.10.257
- [20] Wu SW, Wang G, Wang Q, et al. Enhancement of strength-ductility trade-off in a high-entropy alloy through a heterogeneous structure. *Acta Mater.* 2019;165:444–458. doi:10.1016/j.actamat.2018.12.012
- [21] Li ZM, Pradeep KG, Deng Y, et al. Metastable high-entropy dual-phase alloys overcome the strength-ductility trade-off. *Nature.* 2016;534(7606):227–230. doi:10.1038/nature17981
- [22] Huang HL, Wu Y, He JY, et al. Phase-transformation ductilization of brittle high-entropy alloys via metastability engineering. *Adv Mater.* 2017;29(30):1701678. doi:10.1002/adma.201701678
- [23] Wu XX, Mayweg D, Ponge D, et al. Microstructure and deformation behavior of two TWIP/TRIP high entropy alloys upon grain refinement. *Mater Sci Eng A.* 2021;802:140661. doi:10.1016/j.msea.2020.140661
- [24] Li J, Fang Q, Liu B, et al. Transformation induced softening and plasticity in high entropy alloys. *Acta Mater.* 2018;147:35–41. doi:10.1016/j.actamat.2018.01.002
- [25] Zhang Y, Zhang N, Tang Y, et al. Multiple phase transitions in shock compressed high-entropy alloy Cr₉Mn₉Fe₆₄Co₉Ni₉: experiments and molecular dynamics simulations. *Appl Phys Lett.* 2024;124(10):101901. doi:10.1063/5.0198067
- [26] Agrawal P, Shukla S, Gupta S, et al. Friction stir gradient alloying: A high-throughput method to explore the influence of V in enabling HCP to BCC transformation in a γ -FCC dominated high entropy alloy. *Appl Mater Today.* 2020;21:100853. doi:10.1016/j.apmt.2020.100853
- [27] Chen SY, Tong Y, Tseng KK, et al. Phase transformations of HfNbTaTiZr high-entropy alloy at intermediate temperatures. *Scr Mater.* 2019;158:50–56. doi:10.1016/j.scriptamat.2018.08.032
- [28] Chen JM, Jiang XS, Sun HL, et al. Phase transformation and strengthening mechanisms of nanostructured high-entropy alloys. *Nanotechnol Rev.* 2021;10(1):1116–1139. doi:10.1515/ntrev-2021-0071
- [29] Fang QH, Chen Y, Li J, et al. Probing the phase transformation and dislocation evolution in dual-phase high-entropy alloys. *Int J Plasticity.* 2019;114:161–173. doi:10.1016/j.ijplas.2018.10.014
- [30] Zhao YF, Feng XB, Zhang JY, et al. Tailoring phase transformation strengthening and plasticity of nanostructured high entropy alloys. *Nanoscale.* 2020;12(26):14135–14149. doi:10.1039/d0nr02483j
- [31] Sathiyamoorthi P, Kim HS. High-entropy alloys with heterogeneous microstructure: processing and mechanical properties. *Prog Mater Sci.* 2020;123:100709. doi:10.1016/j.pmatsci.2020.100709
- [32] Wu B, Fu H, Zhou XY, et al. Severe plastic deformation-produced gradient nanostructured copper with a strengthening-softening transition. *Mater Sci Eng A.* 2021;819:141495. doi:10.1016/j.msea.2021.141495
- [33] To S S, Wang H BLW. Materials characterisation and mechanism of micro-cutting in ultra-precision diamond turning. Berlin: Springer; 2018.
- [34] Yuan S, Gan B, Qian L, et al. Gradient nanotwinned CrCoNi medium-entropy alloy with strength-ductility synergy. *Scr Mater.* 2021;203:114117. doi:10.1016/j.scriptamat.2021.114117
- [35] Wu B, Fu H, Luo JS, et al. Excellent strength-ductility synergy of Cu-Al alloy with a gradient nanograined-nanotwinned surface layer. *Mater Sci Eng A.* 2024;901:146524. doi:10.1016/j.msea.2024.146524
- [36] Mordyuk BN, Prokopenko GI, Vasylyev MA, et al. Effect of structure evolution induced by ultrasonic peening on the corrosion behavior of AISI-321 stainless steel. *Mater Sci Eng A.* 2007;458(1–2):253–261. doi:10.1016/j.msea.2006.12.049
- [37] Kahle M, Kleber M, Jahn R. Review of XRD-based quantitative analyses of clay minerals in soils: the suitability of mineral intensity factors. *Geoderma.* 2002;109(3):191–205. doi:10.1016/S0016-7061(02)00175-1.
- [38] Lu L, Sahajwalla V, Kong C, et al. Quantitative X-ray diffraction analysis and its application to various coals.

- Carbon. 2001;39(12):1821–1833. doi:[10.1016/S0008-6223\(00\)00318-3](https://doi.org/10.1016/S0008-6223(00)00318-3).
- [39] Pürstl JT, Jones HO, Edwards TEJ, et al. On the extraction of yield stresses from micro-compression experiments. *Mater Sci Eng A*. 2021;800:140323. doi:[10.1016/j.msea.2020.140323](https://doi.org/10.1016/j.msea.2020.140323)
- [40] Pfetzinger-Micklich J, Ghisleni R, Simon T, et al. Orientation dependence of stress-induced phase transformation and dislocation plasticity in NiTi shape memory alloys on the micro scale. *Mater Sci Eng A*. 2012;538:265–271. doi:[10.1016/j.msea.2012.01.042](https://doi.org/10.1016/j.msea.2012.01.042)
- [41] Zhang YF, Li Q, Gong M, et al. Deformation behavior and phase transformation of nanotwinned Al/Ti multilayers. *Appl Surf Sci*. 2020;527:146776. doi:[10.1016/j.apsusc.2020.146776](https://doi.org/10.1016/j.apsusc.2020.146776)
- [42] Omori T, Ando K, Ohnuma I, et al. BCC-HCP-FCC multiple transformations and ϵ loop in the Fe-Cr-Co-Mn system. *J Phase Equilib Diff*. 2021;42(5):735–747. doi:[10.1007/s11669-021-00901-0](https://doi.org/10.1007/s11669-021-00901-0)
- [43] Wang B, Urbassek HM. Phase transitions in an Fe system containing a bcc/fcc phase boundary: An atomistic study. *Phys Rev B*. 2013;87(10):104108. doi:[10.1103/PhysRevB.87.104108](https://doi.org/10.1103/PhysRevB.87.104108)
- [44] Wu H, Fan G. An overview of tailoring strain delocalization for strength-ductility synergy. *Prog Mater Sci*. 2020;113:100675. doi:[10.1016/j.pmatsci.2020.100675](https://doi.org/10.1016/j.pmatsci.2020.100675)
- [45] Zhang LJ, Yu PF, Fan JT, et al. High pressure induced the polymorphism phase transition in the Fe₄₀Mn₄₀Co₁₀-Cr₁₀ multi-principal element alloy. *Intermetallics*. 2021;136:107268. doi:[10.1016/j.intermet.2021.107268](https://doi.org/10.1016/j.intermet.2021.107268)
- [46] Kaushik L, Kim MS, Singh J, et al. Deformation mechanisms and texture evolution in high entropy alloy during cold rolling. *Int J Plast*. 2021;141:102989. doi:[10.1016/j.ijplas.2021.102989](https://doi.org/10.1016/j.ijplas.2021.102989)
- [47] Chandan AK, Tripathy S, Sen B, et al. Temperature dependent deformation behavior and stacking fault energy of Fe₄₀Mn₄₀Co₁₀Cr₁₀ alloy. *Scr Mater*. 2021;199:113891. doi:[10.1016/j.scriptamat.2021.113891](https://doi.org/10.1016/j.scriptamat.2021.113891)
- [48] Efe M, Moscoso W, Trumble KP, et al. Mechanics of large strain extrusion machining and application to deformation processing of magnesium alloys. *Acta Mater*. 2012;60(5):2031–2042. doi:[10.1016/j.actamat.2012.01.018](https://doi.org/10.1016/j.actamat.2012.01.018)
- [49] Guo Y, Saldana C, Dale Compton W, et al. Controlling deformation and microstructure on machined surfaces. *Acta Mater*. 2011;59(11):4538–4547. doi:[10.1016/j.actamat.2011.03.076](https://doi.org/10.1016/j.actamat.2011.03.076)