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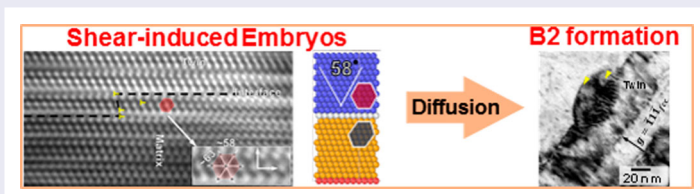
Deformation induced intermediate metastable lattice structures facilitate ordered B2 nucleation in a fcc-based high entropy alloy

Deep Choudhuri ^{a,b}, Shivakant Shukla ^b, Bharat Gwalani ^{a,b}, Rajarshi Banerjee ^{a,b} and Rajiv S. Mishra ^{a,b}

^aAdvanced Materials and Manufacturing Processes Institute, University of North Texas, Denton, TX, USA; ^bDepartment of Materials Science and Engineering, University of North Texas, Denton, TX, USA

ABSTRACT

Ordered B2 precipitates typically nucleate at the grains-boundaries of fcc-based high entropy alloys. Here, we report a novel mixed-mode coupled displacive-diffusional transformation resulting in homogeneously distributed intra-granular B2 precipitates within the fcc matrix. Severe plastic deformation forms compositionally invariant, metastable distorted fcc structures, resembling hexagon-like templates, at the deformation twin-boundaries. These shear-induced hexagon-like templates correspond to the symmetry of the $\{111\}_{bcc}$ planes, and act as sites for B2 nucleation, establishing the fcc-bcc Kurdjumov–Sachs (KS) orientation relationship. However, the composition of these B2 precipitates is far-from-equilibrium. Subsequent isothermal annealing causes solute partitioning driving the composition of the B2 precipitates towards equilibrium.



IMPACT STATEMENT

For the first time, a mixed mode displacive-diffusional fcc-to-bcc-ordered B2 transformation mechanism was revealed in a deformed fcc-based $\text{Al}_{0.3}\text{CoCrFeNi}$ complex concentrated or high entropy alloy.

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KEYWORDS

High entropy alloys or Complex concentrated alloys; Bcc-ordered B2 precipitation; Structural models; Transmission electron microscopy; Deformation twinning

The ordered body-centered-cubic B2 intermetallic phase in aluminides such as Ni–Al and Co–Al is well known for producing high-temperature strength [1–5]. To increase their workability, ternary alloys including Co–Al–Ni were later developed, where the microstructure contained plastically softer disordered-fcc phase and harder B2 [2,3]. In fact, fcc-B2 microstructures are now regularly observed in complex concentrated alloys (CCAs) or high entropy alloys, and offer promising mechanical properties; e.g. excellent strength-ductility combination, fatigue resistance, etc. [6–11]. Together, the combined body of literature indicated that fcc-B2 microstructures may be desirable for structural applications, and mandates investigation of transformation mechanisms that facilitate bcc-ordered B2 formation within an fcc-matrix.

Recently, a detailed investigation of phase stability regimes in the fcc-based $\text{Al}_{0.3}\text{CoCrFeNi}$ CCA [10], via microstructural characterization and thermodynamic calculations, has demonstrated that the B2 phase has a very high nucleation barrier, due to the high fcc/B2 interfacial energy. Consequently, despite the high driving force for B2 precipitation, it is very difficult to homogeneously nucleate B2 precipitates within the fcc-matrix, resulting in heterogeneous nucleation of B2 precipitates at the fcc grain boundaries [10,11]. However, cold rolling and subsequent annealing of the $\text{Al}_{0.3}\text{CoCrFeNi}$ CCA can produce a homogeneous dispersion of B2 precipitates within the fcc-matrix [11]. The prior cold working introduces a high density of defects such as dislocations and deformation twins, and the B2 precipitates form at the interfaces between the deformation twins

CONTACT Deep Choudhuri deep.choudhuri@unt.edu, deep.choudhuri@gmail.com Advanced Materials and Manufacturing Processes Institute, University of North Texas, Denton, TX 76207, USA; Department of Materials Science and Engineering, University of North Texas, Denton, TX 76207, USA

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and the fcc-matrix as well as at twin–twin intersections [11]. These B2 precipitates exhibited both Kurdjumov–Sachs (KS) and Nishiyama–Wassermann (NW) orientation relationships (ORs) with the fcc matrix [11].

The interface between a deformation twin and the fcc-matrix invariably contain stacking faults (SFs) arising from $1/6 \langle 112 \rangle$ Shockley partials, and local fcc-lattice distortion [12]. In general, dislocation assisted heterogeneous phase nucleation is well known [13], but such mechanism does not satisfactorily account for the observed precipitate–matrix OR. On the other hand, local distortion of the parent lattice can partially acquire the crystallographic symmetry of the daughter phase. An example of such lattice distortion can be gleaned from the Borger–Burger–Olsen–Cohen model, which was originally proposed to explain diffusionless bcc martensitic transformations in fcc-based alloys; e.g. austenitic steels [14,15]. Their hard-sphere geometric model describes the transformation as an application of simple shear to an ABCABC stack of $(111)_{fcc}$ planes; i.e. Thompson’s tetrahedron. Here, application of shear strain corresponding to $1/\sqrt{2}$ causes the tetrahedron to develop a twin orientation relative to the original configuration. However, at intermediate strains, the fcc tetrahedron acquires bcc-like ‘intermediate structures’ that manifested KS and NW ORs [14,15]. Therefore, we have attempted to identify contributions from stacking faults and local fcc-lattice distortion on fcc–B2 transformation by examining the microstructures in the deformed condition and early stages of B2 precipitation in a prior cold-rolled $\text{Al}_{0.3}\text{CoCrFeNi}$ CCA.

Experiments were performed using rectangular strips of $\text{Al}_{0.3}\text{CoCrFeNi}$ CCA, of composition 6.69%Al–23.14%Co–23.36%Cr–23.43%Fe–23.38%Ni (at.%), which were first homogenized at 1150°C for 1H, and subsequently cold-rolled to 50% reduction in thickness. The cold-rolled pieces were then annealed at 800°C for 30 mins (0.5H) and 50H to compare the early and late stages of B2 formation, respectively. The thermodynamic basis for selecting the homogenizing and annealing temperatures is provided elsewhere [10,11]. Microstructural characterization was carried out using FEI’s Nova NanoLab 200TM scanning electron, and FEI Tecnai F20-FEGTM (operating at 200 kV) transmission electron microscopes.

The $[011]_{fcc}$ selected area diffraction pattern (SADP) recorded from the 50% cold-rolled (CR) condition (Figure 1(a)), shows reciprocal lattice reflection from multiple variant of twins in addition to the primary fcc reflections. (One twin variant is marked with a yellow rectangle). The corresponding dark-field TEM (DFTEM) in Figure 1(b) shows the deformation twins. Backscattered secondary electron (BSE) images presented in Figure 1(c,d) depicts the precipitation inside the

fcc-matrix after subsequent annealing at 800°C for 0.5H and 50H, respectively. We will first focus on the TEM observations from the cold-rolled microstructure, which revealed two key features. First, in many cases the longer edge of the twins forms small angles, i.e. $\sim 2\text{--}5^\circ$ (see Figure 1(b)), with respect to the expected $\{111\}_{fcc}$ habit plane [11]. Second, the fcc reciprocal lattice reflections in Figure 1(a) exhibit azimuthal or ‘side-ways’ streaking, which is comparable to fcc-microstructures that had experienced significant shear deformation [16]. Together, these observations suggest local lattice misorientations, and their implication on B2 formation will be discussed later in the context of early stages of precipitation.

The BSE images presented in Figure 1(c,d) show the change in microstructures after annealing at 0.5H and 50H. The BSE in Figure 1(d) clearly resolved B2 plates after 800°C/50H, and the presence of the B2 phase was confirmed via $[001]_{B2}$ superlattice reflections in the $[011]_{B2}$ micro-diffraction pattern (inset of Figure 1(c)) (also see supplementary Figure S1(a)). We emphasize that past studies had indicated that such B2 plates at 800°C/50H exhibited both Kurdjumov–Sachs (KS) and Nishiyama–Wassermann (NW) ORs. (See supplementary Figure S1 and [11].) In stark contrast to the 800°C/50H condition, the BSE of 800°C/0.5H does not clearly reveal B2 precipitates (Figure 1(c)), since the microstructure appears to be dominated with channeling contrast arising from deformation experienced by the prior cold rolling step. Therefore, B2 precipitation at 800°C/0.5H was examined using transmission electron microscopy in detail.

A bright-field TEM (BFTEM) in a TEM foil, plucked from the 800°C/0.5H condition, is shown in Figure 2(a). The BFTEM recorded by using a two-beam condition using $\mathbf{g} = [\bar{1}1\bar{1}]_{fcc}$ near the $[011]_{fcc}$ zone axis revealed several B2 precipitates ‘wetting’ the edges of an extended deformation twin. (Also see supplementary Figure S2(a).) The large dimensions of these B2 precipitates ($\sim 10\text{--}20$ nm) suggested fairly fast kinetics at 800°C/0.5H. A $[011]_{fcc}$ SADP was recorded from this region by placing the selected-area aperture covering B2, twin and the parent fcc-matrix (SADP presented as inset in Figure 2(a)). The SADP revealed that, at the early stages of precipitation, the B2 phase shares a strict KS orientation relationship (OR) with the twin and the parent fcc phase; i.e. $(011)_{B2} // (111)_{fcc\text{--matrix \& twin}}$ and $[11\bar{1}]_{B2} // [0\bar{1}1]_{fcc\text{--matrix \& twin}}$. This B2–fcc KS OR is retained even after long-term annealing at 800°C/50H (see ref# [11] and supplementary Figure S1(b)). In one case, NW OR; i.e. $(001)_{B2} // (0\bar{1}1)_{fcc}$ and $[110]_{B2} // [111]_{fcc}$, was also noted in the TEM foil extracted from the 800°C/0.5H condition. (See supplementary Figure S3). The imaging condition used to acquire Figure 2(a) also

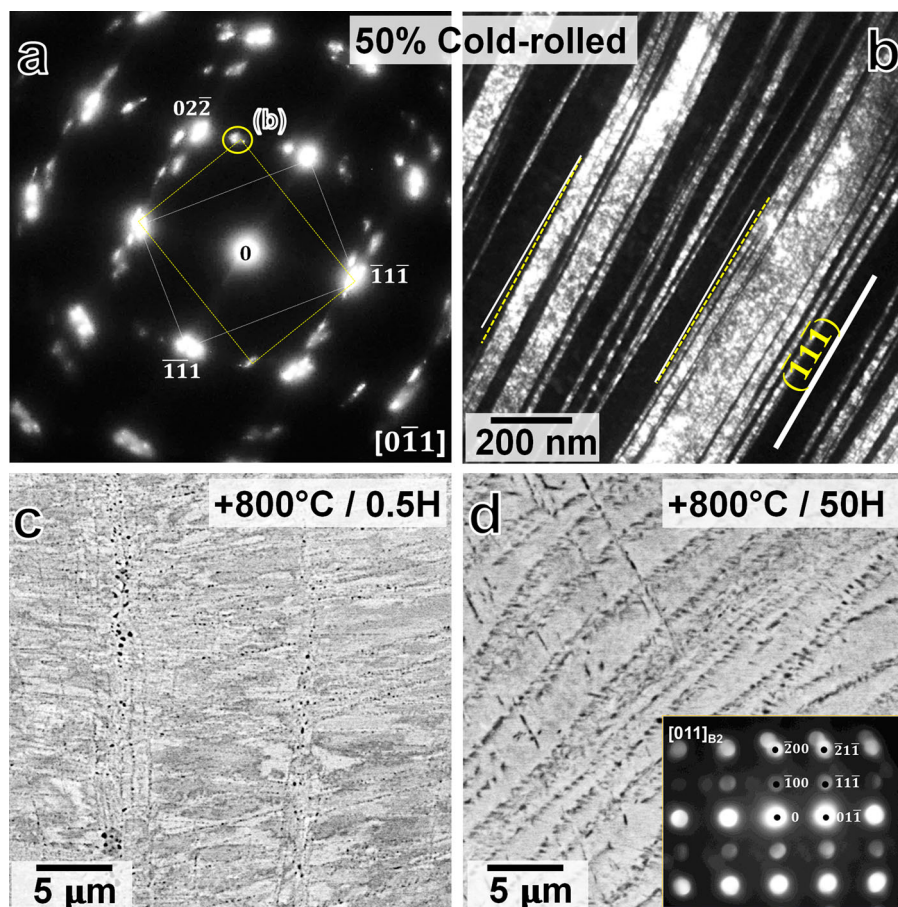


Figure 1. TEM of the cold-rolled condition: (a) SADP pattern recorded along the $[011]_{fcc}$ zone axis, and (b) the corresponding dark-field TEM (DFTEM) showing deformation twins. BSE of microstructures after annealing at 800°C for (c) 0.5 H (30 mins) and (d) 50H. The inset in (d) shows $[011]_{B2}$ micro-diffraction pattern.

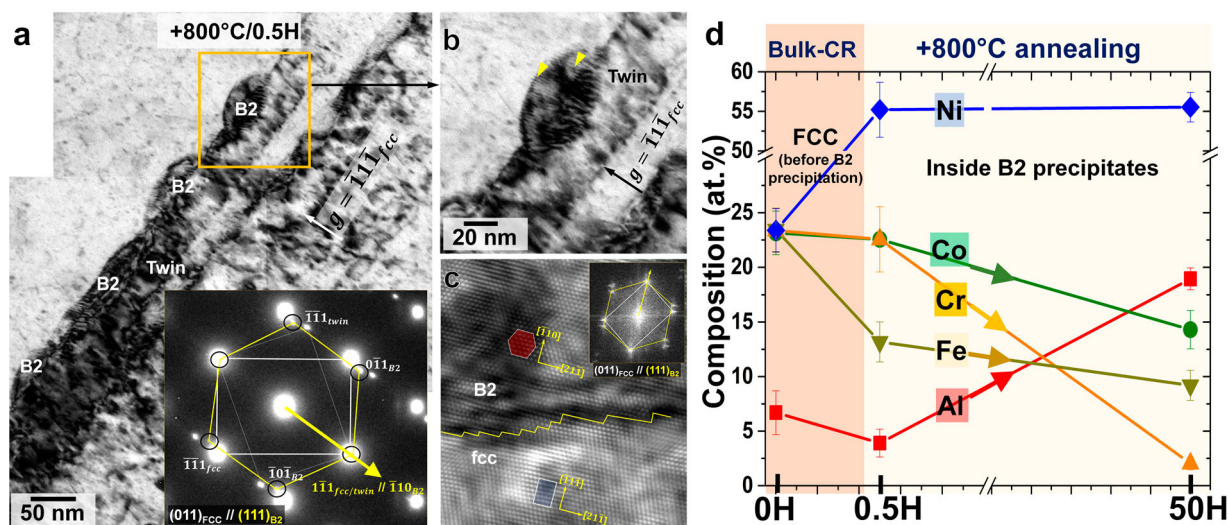


Figure 2. Microstructural characterization after annealing at 800°C . (a) bright-field TEM (BFTEM) image showing B2 precipitation along twin-matrix interfaces. The inset shows the $[011]_{fcc}$ SADP with the fcc -B2 Kurdjumov–Sachs (KS) orientation relationship. (b) A magnified BFTEM showing fcc -B2 interface. (c) HRTEM of the same fcc -B2 KS interface and the corresponding fast Fourier transform (FFT). In the FFT, $[111]_{B2}$ and $[011]_{fcc}$ zone axes are indicated with a yellow hexagon and white rectangle, respectively. (d) STEM-EDS measurements comparing elemental concentrations in the fcc -matrix of the cold-rolled (Bulk-CR) condition prior to B2 precipitation, and elemental concentrations B2 in after 0.5 and 50H of annealing.

revealed parallel fringes were also observed at one of the interfaces between B2 and the parent *fcc*-matrix (rectangle in Figure 2(a)), which is also shown in a magnified view in Figure 2(b). (Also see supplementary Figure S2(b)). Such interfacial ‘structure’ was examined by performing high-resolution TEM (HRTEM) on a thin section containing a B2/*fcc* KS interface; i.e. *fcc* and B2 are oriented along $[011]_{fcc}$ and $[111]_{B2}$, respectively. The HRTEM presented in Figure 2(c) shows that the B2/*fcc* interface is comprised of ledges and steps (marked with lines), where the steps lie parallel to $(111)_{fcc}$ planes. Further analysis of the B2/*fcc* KS interfacial structure is progress. Regardless, the B2 precipitates form a semi-coherent interface with the parent *fcc* lattice at a very early stage of its formation (i.e. 30 mins of annealing at 800°C).

Phase compositions of the early (0.5H) and late (50H) stages of B2 were further measured and compared with the cold rolled (CR) condition using scanning transmission electron microscopy energy dispersive X-ray spectroscopy (STEM-EDS). Figure 2(d) shows significant differences in B2 composition after 800°C/0.5H and 800°C/50H conditions. (Also see supplementary Figure S(4) and Supplementary Table I). The CR condition did not reveal any detectable compositional partitioning (supplementary Figure S4), nor was any prior B2 precipitation noted from diffraction patterns (e.g. Figure 1(a)). STEM-EDS plot presented in Figure 2(d) has two key implications. First, the B2 formed at the early stages; i.e. 800°C/0.5H, has a non-equilibrium composition compared to the late stages of annealing. Note the differences in Cr, Co, Al and Fe concentrations in B2 800°C/0.5H and 800°C/50H. This is rather surprising because intermetallic compounds typically nucleate with the equilibrium composition [17]. In other words, the *fcc*-B2 transformation in CCAs cannot be described on the basis of classical nucleation, where the daughter phase nucleates with equilibrium composition [13]. Second, in the 800°C/0.5H specimen, the concentrations of Co, Cr, and Al remains comparable to the bulk composition of the CR specimen (prior to annealing and B2 precipitation). Although, we observe that Ni has substantially partitioned in to B2, while Fe has an opposite trend. Regardless, the nearly unchanged Co, Cr and Al concentrations in the two CR and 800°C/0.5H conditions suggested a compositionally invariant metastable *intermediate structure* formation from severe plastic deformation, i.e. 50% cold-rolling, where the *intermediate structures* have not fully acquired the *bcc* symmetry of the B2 phase. Subsequent annealing causes Ni enrichment (and Fe depletion) within such structures and stabilization of the B2 phase at 800°C/0.5H. In other words, twin-assisted B2 formation mechanism may be described via a mixed mode

mechanism comprising a displacive structural transformation coupled with solute diffusion.

Fast kinetics associated with B2 precipitation did not permit identification of such metastable intermediate structures in the 800°C/0.5H condition. Therefore, we have conducted a detailed analysis of the twin-matrix interfaces in the CR microstructure; by taking into account that local lattice deformation can form intermediate structures, e.g. Borger–Burger–Olsen–Cohen model [14,15]. Figure 3(a) shows the HRTEM of a deformation twin-matrix interface. The FFTs in Figure 3(b) depict regions at the longer edge of the twin-matrix interface, while Figure 3(c,d) show regions inside the twin and parent lattice, respectively, that were nominally away from the interface. Also, in Figure 3(b), the twin-matrix interface was approximately demarcated with a dotted black line. (Qualitatively, it appears that the twinned region had experience more distortion compared to the parent lattice). We note that the interface has a ledge-like feature that appears to have been formed by the stacking of four $1/6 < 112 >$ partial dislocations. It is likely that the formation of such ledges caused the longer edge of the twins to deviate from the $\{111\}$ habit plane (Figure 1(a)).

Importantly, near that ledge shown in Figure 3(b) is a row of local ‘pseudo-hexagonal’ structures (indicated with red-shaded hexagons), which are also shown as an inset on the top-right of Figure 3(b). These structures are termed pseudo-hexagons because their angles deviated by $\sim 2\text{--}3^\circ$ from the ideal hexagon angle of 60° , but are substantially different from those in the $(011)_{fcc}$ motifs seen in Figure 3(c,d) (indicated with blue-shaded ‘squished’ hexagons). Furthermore, the orientation of these pseudo-hexagons corresponds to the orthogonal $[11\bar{1}]_{fcc}$ and $[2\bar{1}\bar{1}]_{fcc}$ directions (also corresponds to KS-OR) of both twin and parent matrices. By comparing with the wetting layer of B2 at the twin-matrix interface in 800°C/0.5H (BFTEM in Figure 2(a)), we surmised that, in the twinned condition, the pseudo-hexagons may have provided precursory lattice templates for (111) planes of B2, which, after annealing, acquires a B2/*fcc* KS orientation.

To better understand the role of the intermediate structures on B2 formation, we have considered two geometric hard sphere models comprising of ABCABC stacking of $(111)_{fcc}$ planes. Here, the same hard sphere model was constructed using the OVITO software [18], which, also, can impose predefined simple shear and simultaneously calculate the local coordination; i.e. *fcc*, *bcc* or *hcp*, via the common neighbor algorithm (CNA) [18]. A rectangular box, comprising of hard spheres arranged in an *fcc* lattice, was constructed with the orthogonal axes along the $[111]_{fcc}$, $[\bar{1}01]_{fcc}$ and $[\bar{1}2\bar{1}]_{fcc}$ (Figure 4a1–a2 and Figure 4b1). The *first* model involves

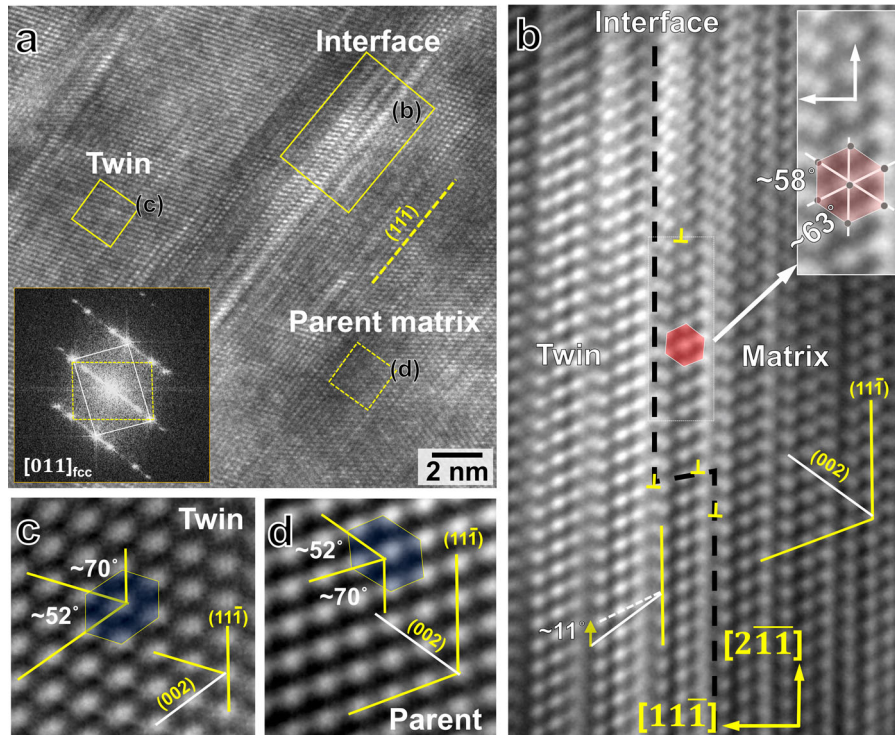


Figure 3. HRTEM of a twin along $[011]_{fcc}$ in the cold-rolled condition. (a) Raw HRTEM image of a twin and the corresponding FFT as inset. (b) FFT of a HRTEM showing the twin-matrix interfacial region. The small arrow in the bottom left of panel '3b' indicates distortion of the (002) plane by $\sim 11^\circ$ angle inside the twin. Here, the dotted white line shows the trace of distorted (002) plane and the bold white line is the (002) trace inside the twinned region far from the interface. Reference *fcc* lattices inside the (c) twin and (d) parent regions away from the twin-matrix interface is also shown.

adapting the Borger–Burger–Olsen–Cohen model where one applies simple shear to the hard sphere collective or lattice (Figure 4(a)). The *second* approach involves shearing of individual $(111)_{fcc}$ planes (Figure 4(b)); instead of the entire lattice, i.e. stacking fault formation.

First model—Figure 4(a) shows the two perspective views when viewed along the $[111]_{fcc}$ (panel a1) and $[\bar{1}01]_{fcc}$ (panel a2). Simple shear was applied to the top-half of the model along $[\bar{1}2\bar{1}]_{fcc}$ until either the twinning shear was achieved, or a twin interface develops; i.e. $\gamma = 1/\sqrt{2} = 0.707$ (panel a5 and a8). The sheared structures were categorized according to the (i) shear strain values of $\gamma = 0.29$ (a3 & a6), $\gamma = 0.35$ (a4 & a7) and $\gamma = 0.71$ (a5 & a8), and (ii) viewing directions perpendicular to two conjugate $\{110\}$ planes; i.e. $(\bar{1}10)$ (red line and arrow in a1) and $(\bar{1}01)_{fcc}$ (black line and arrow in a1), which are presented as top (a3, a4 and a5) and bottom (a6, a7 and a8) rows of the panels. At intermediated strains, i.e. $\gamma = 0.29$ and 0.35 , the *fcc* lattice acquires a bcc-like coordination (based on CNA analysis [18]), and $\gamma = 0.71$ causes twin formation. Here, on the conjugate $(\bar{1}10)$ plane (a3 and a4), the angle between the prior $\{111\}_{fcc}$ planes approaches 60° (i.e. 53° and 58°) at intermediate shear strains. The hexagonal templates are also seen at $\gamma = 0.35$, whose angular separations resemble

the HRTEM observations of the twin-matrix interfacial regions (Figure 3(b)). In relation to the experimental observations, the hard-sphere model indicates that lattice shear, presumably caused by the pile of dislocations near the twin-matrix, can form pseudo-hexagonal templates (Figure 3(b)), which are precursors to KS OR between *fcc* and B2 precipitate.

Interestingly, the same shear strain along $[\bar{1}2\bar{1}]_{fcc}$ causes the bcc-like region to acquire rectangular templates on the $(\bar{1}01)$ plane (bottom row of a6 and a7), whose crystallographic directions (with respect to *fcc*) corresponded to NW OR. Although the rectangular template was not evident in the HRTEM of the CR condition, the presence of *fcc*-B2 NW OR at $800^\circ\text{C}/0.5\text{H}$ and $800^\circ\text{C}/50\text{H}$ conditions validate the model. (See supplementary Figures S1 and S3, and [12].)

Second model—Figure 4(b) shows the change in angles after introducing SFs on alternate $(111)_{fcc}$ planes; marked '1' and '2' in panel b1. Note the SF was not introduced between '1' and '2' because that would have created a 'hcp-like' phase; which is not observed in our TEM observations. After introducing the first SF the lattice angles correspond to the *fcc* structure (panel b2). However, the region between SF-1 and SF-2 in panel b3 appears to acquire an angle closer to that of $(111)_{bcc}$,

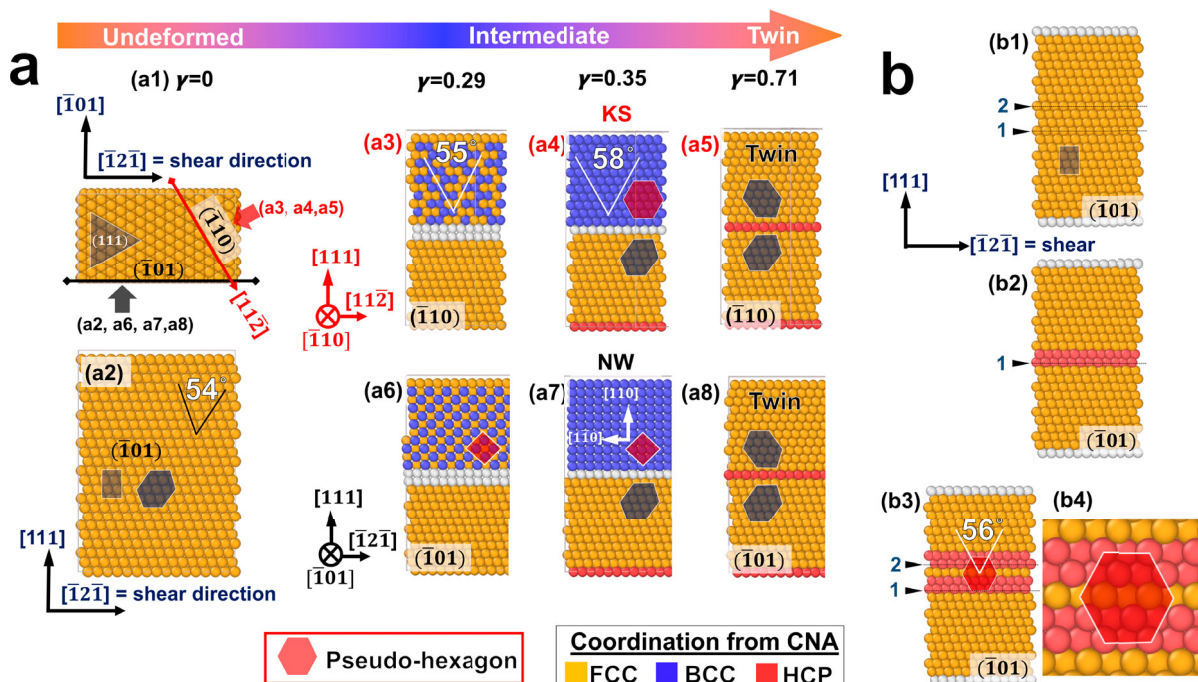


Figure 4. Two hard sphere models: (a) Borger–Burger–Olsen–Cohen model—(a1) and (a2) show two views of the undeformed *fcc* lattice, while (a3, a6), (a4, a7) and (a5, a8) show the same lattice after experiencing increasing shear strains. The top and bottom rows in panels (a3)–(a5) and (a6)–(a8) represent two views that are perpendicular to two conjugates $\{110\}$; as indicated in (a1), respectively. Note that panels (a3, a6) and (a4, a7) present the intermediate stages of shear where ‘bcc-like’ structures are observed, while (a5, a8) depicts the final stage corresponding to a twin. (b) Model showing local lattice deformation as due to stacking faults. Note that the CNA analysis of the shear structure also shows the resulting microstructure in three different colors, orange (*fcc*), blue (*bcc*) and red (*hcp*).

i.e. 56° . In other words, at least two SFs are required to form a $(111)_{bcc}$ template (highlighted with a magnified view in panel b4). The key factor that differentiates the second model from Borger–Burger–Olsen–Cohen model is the lack of ‘bcc’ coordination in the former, (e.g. in panels a4 and a7 in Figure 4(a) the bcc coordination is represented with blue colored atoms). Moreover, Borger–Burger–Olsen–Cohen model elegantly accounts for the both KS and NW ORs for a given shear strain; seen in our TEM observations. However, it is suspected that the two models may represent extreme situations, and in practice the likely lattice displacement, leading to $(111)_{bcc}$ templates, is expected to be a combination of both models.

The mechanism of *fcc* to *bcc*-based ordered B2 transformation was examined by annealing 50% cold-rolled, *fcc*-based $\text{Al}_{0.3}\text{CoCrFeNi}$ CCA at 800°C for 0.5H and 50H. Experimental evidence involving detailed TEM of the microstructure at early stages of B2 precipitation was coupled with qualitative hard sphere models involving two types of deformation modes. Our analysis revealed that the *fcc*-to-B2 transformation can be classified as a mixed mode type transformation; comprising a displacive structural component coupled with solute diffusion. Plastic deformation of the *fcc* lattice of this CCA forms compositionally invariant metastable intermediate

structures, which contain structural motifs resembling the *bcc* symmetry or crystallography, e.g. $(111)_{bcc}$ templates required for KS oriented B2 nucleation (Figures 3(b) and Figure 4). Subsequently, annealing at 800°C partitions solute into the regions containing the metastable structures and causes B2 phase formation (Figure 2(a)). B2 composition at early stages, 0.5H, was far from equilibrium, and near-equilibrium composition after annealing for longer duration; i.e. $800^\circ\text{C}/50\text{H}$ (Figure 2(d)).

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ORCID

Deep Choudhuri <http://orcid.org/0000-0001-9590-9855>
Rajiv S. Mishra <http://orcid.org/0000-0002-1699-0614>

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