Nano-twin Mediated Plasticity in Carbon-containing FeNiCoCrMn High Entropy Alloys

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Abstract

Equiatomic FeNiCoCrMn alloy has been reported to exhibit promising strength and ductility at cryogenic temperature and deformation mediated by nano-twinning appeared to be one of the main reasons. We use the FeNiCoCrMn alloy as a base alloy to seek further improvement of its mechanical properties by alloying additional elements, i.e., interstitial carbon. The effects of carbon on microstructures, mechanical properties and twinning activities were investigated in two different temperatures (77 and 293 K). With addition of 0.5 at.% C, the high entropy alloy still remains entirely single phase face-centered cubic (FCC) crystal structure. The materials can be cold rolled and recrystallized to produce a microstructure with equiaxed grains. Both strain hardening rate and strength are enhanced while high uniform elongations to fracture (~ 70% at 77 K and ~ 40% at 293 K) are still maintained. The increased strain hardening and strength could be caused by the promptness of deformation twinning in C-containing high entropy alloys.

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1. Introduction

Most traditional alloys, such as steels, brasses and bronzes, aluminum alloys, magnesium alloys and Ni base superalloys, are mainly based on one principle element with addition of minor alloying elements. Recently, an interesting subset of compositionally complex alloys is emerging in which the constituent elements are present in equal- or near-equal-atomic concentrations and therefore “solvent” or “solute” cannot be distinguished [1-13]. Numerous experimental and theoretical investigations have shown that, contrary to the general physical metallurgy intuition (e.g., increasing number of additive elements with very different crystal structures will simultaneously increase the probability that second phases will form) and traditional notions of solid solubility based on empirical rules such as Hume-Rothery, these alloys containing multiple constituent elements in equiatomic or near-equiatomic concentrations tend to crystallize as solid solutions with simple (FCC or BCC) crystal structures [3, 4]. One example is equiatomic FeNiCoCrMn alloy, which exhibit single-phase with FCC crystal structure [5-9]. Since its first exploration in 2004 [5], high thermodynamic stability [6], excellent malleability [7], and slow diffusion [10] have been reported for this alloy. Recent mechanical investigations found that this alloy had both practical and scientific interests from several perspectives. Its yield strength showed an intermediate temperature-dependence that is between pure FCC and BCC metals [11, 12]. Moreover, contrary to most traditional materials in which strength and ductility shows an inverse temperature-dependence, a remarkable increase in strength accompanied by a substantial increase in ductility with decreasing deformation temperature from 293 to 77 K was observed in this alloy [12]. It also holds remarkable fracture toughness at 77 K, as high as $200 \text{ MPa}\cdot\text{m}^{1/2}$, which is comparable to the very best cryogenic steels (e.g., austenitic stainless steels and

high-Ni steels) [13]. The outstanding mechanical properties in cryogenic temperature accompany with the observation of nano-twinning formation during plastic deformation.

The nano-twinning observation is reminiscent of another group alloys - Twinning-Induced-Plasticity (TWIP) steels which have been widely used in automotive industry since its first development of high manganese austenitic steels by Hadfield in 1888 [14]. TWIP steels were found to hold high tensile strength with extraordinary ductility [15-18]. The characterizations of the deformation-microstructure-mechanical property relationship of compositionally different TWIP steels have reasonably attributed the observed promising mechanical properties to the formation of nano-scaled deformation twins which will significantly raise the strain hardening by serving as obstacles to dislocation motion and subdivisions of initial grains into twin-surrounded lamellas [19-22].

Usually in TWIP steels, alloying additions of C, Si and/or Al are needed to obtain the ideal combination of high strength and good uniform elongation due to their capability for triggering nano-twinning in Fe-Mn TWIP steels [23]. Therefore, a similar design concept is used here to seek further improvement of the mechanical properties of FeNiCoCrMn high entropy alloys by alloying. As the first step, carbon will be alloyed to the equiatomic FeNiCoCrMn alloy to investigate its potential of enhancing the mechanical performance. In TWIP steels, the carbon concentration will be normally maintained below 0.6 at.% in order to obtain a carbide-free, fully austenitic microstructure with lowered stacking fault energy which is uniquely important for the formation of deformation twinning [23]. Thus in the current study, 0.5 at.% carbon will be added and its effects on the microstructure and mechanical properties of the FeCrNiCoMn high entropy alloys will be investigated.

2. Experimental Methods

Equiatomic FeNiCoCrMn alloys with the addition of 0.5 at.% C was produced by arc melting the elements Fe, Ni, Co, Cr, Mn (>99.9% pure) and graphite powder in a water-cooled copper crucible under Ar atmosphere. Rectangular ingots measuring 12.7 mm × 12.7 mm × 127 mm were produced by drop-casting the melts into copper molds. The ingots were homogenized for 24 hours at 1473 K, followed by an air-cooling. They were then cold rolled along the longitudinal ingot direction to a total thickness reduction of ~86% without intermediate annealing. Flat dog-bone-shaped specimens with a gage length of 10 mm were cut from the cold-rolled sheets with their longitudinal axes perpendicular to the rolling direction followed by recrystallized at 1100 °C for 1 hour. Before the tensile tests, all faces of sample gage sections were ground through 600-grit SiC paper and nine Vickers microhardness indents spaced 1 mm apart were made along the gage lengths using a LECO LM 100_{AT} Vickers Hardness tester with a force of 200 g. These pre-existing indents will be used to identify the uniform elongations to fracture (ϵ_f) by averaging the change in the distance between adjacent indents. Tensile tests were performed with a screw-driven mechanical testing machine (Instron) at an engineering strain rate of 10^{-3} s^{-1} and temperatures of 77 and 293 K. For each temperature, three specimens were conducted to rupture to obtain the stress-strain curve, and four specimens were tensile tested and stopped at strains of 6.5%, 13%, 19.5%, 32.5% to study the microstructural evolution during plastic deformation. X-ray diffraction was performed on the gage sections of fractured specimens to determine whether any phase transformation or second phase precipitation had occurred.

Hitachi S4800 field emission gun (FEG)-SEM and 6500 F JEOL FEG-SEM equipped

with a TSL OIM EBSD system were used to characterize the microstructures of the as-annealed and deformed materials. The EBSD measurements were performed with an electron probe current of 3.0 nA at an acceleration voltage of 20 kV, with step size of 20 nm (high magnification) or 300 nm (low magnification). TSL OIM V7TM software was used for qualification and analysis of the EBSD results. EBSD maps are displayed as inverse pole figure (IPF) maps in the direction of the rolling direction (for the initial microstructure) or tensile axis (for the tensile-deformed microstructure). Data "cleaning" procedures [24] were not used.

3. Results

The microstructure of FeNiCoCrMn-0.5C sample annealed at 1100 °C for 1 hour after cold rolling is shown in Fig. 1a, which is electron backscatter diffraction (EBSD) inverse pole figure (IPF) map. Only the FCC solid solution phase was found. As can be seen in the figure, the annealed material contains homogeneous equiaxed grain microstructure without strong texture. Similar to the equiatomic FeNiCoCrMn alloy [12], annealing twins are visible in many grains in the carbon-contained alloys and the thickness of the annealing twin is in the tens μm scales. The average grain size without twin boundaries determined by a linear intercept method was found to be $\sim 115 \mu\text{m}$. The FCC single-phase structure was revealed by X-ray diffraction to remain stable during deformation at room temperature and liquid nitrogen temperature: no evidence of phase transformation or second-phase precipitation was detected after mechanical testing.

Figure 1b shows representative engineering stress vs. plastic strain curves of the

FeNiCoCrMn-0.5C alloy tested at both temperatures; and for comparison, the curves of FeNiCoCrMn alloy with similar grain size is also included in the figure [12]. The 0.2% offset yield strengths (σ_y), ultimate tensile strengths (UTS), uniform elongations to fracture (e_f) and extent of work hardening ($\Delta\sigma = \text{UTS} - \sigma_y$) were summarized in Table I. Similar to the equiatomic FeNiCoCrMn alloy; both strength and ductility increase significantly when the deformation temperature decreases from 293 to 77 K. Carbon additions significantly increase yield strength and UTS of the materials with the uniform elongations to fracture remain high values of $\sim 70\%$ and $\sim 40\%$ at 77 and 293 K, respectively. For example, by adding 0.5 at.% carbon, the 77 K-UTS is increased by 33% from 820 to 1090 MPa; and the 293 K-UTS is increased by 26% from 520 to 655 MPa. One distinct feature that can be immediately seen from the stress-strain curves (Fig. 1b) is the largely enhanced average slope of the plastic region, which could be an indication of the average strain hardening rate, for the carbon-containing alloy compared to that for the carbon-free FeNiCoCrMn alloy.

The EBSD maps showing the microstructures of the FeNiCoCrMn-0.5C alloy tensile-deformed to different engineering strains (6.5%, 13%, 19.5%, 32.5%) were shown in Figure 2. The grains still remain equiaxed when the strain are small (6.5% or 13%) and become elongated along the tensile direction with further straining. The initial material (Fig. 1a) shows nearly equiaxed grain structures. As plastic deformation occurs in the grains, orientation fluctuations appear quickly, even after only 6.5% strain (Fig. 2a-2b), and these in-grain orientation variations increase with increasing strain (Fig. 2c-2j), indicating that plastic tensile deformation generated dislocations and caused these misorientations. The range of crystal orientation within a grain increases with the level of

the plastic deformation due to the increased density of the stored dislocations [25]. Another feature is that with increasing strain, the grains are highly distorted and exhibit orientations mainly with the $\langle 1\ 1\ 1 \rangle$ - or $\langle 1\ 0\ 0 \rangle$ - crystallographic direction parallel to the tensile axis.

For the FeNiCoCrMn-0.5C alloy, when it was deformed at 77 K, at an engineering strain of 6.5%, near-equally spaced individual twins with a width of ~ 10 nm were revealed by back-scattered SEM (Fig. 3). These twins nucleated at grain boundaries and do not extend to the opposite grain boundary, but only grow partially into the grain interior, without impinging on other interfaces. A magnified view of these areas shows that some of the twins will be bundled and form twin bands with ~ 100 nm thickness. It is noted that failure to reveal the existence of deformation twin (bands) using EBSD (Fig. 2a) could be caused by the low density of mechanical twins at this level of strain and a much smaller width of individual twins (~ 10 nm) and thickness of the twin bundle (~ 100 nm) compared to the EBSD scanning step size (300 nm). At 13% engineering strain, a few band-like features in some $\langle 1\ 1\ 1 \rangle$ //TA ($\langle 1\ 1\ 1 \rangle$ direction aligned to the tensile axis) oriented grains (Fig. 2c) were observed. To have a better visualization of these bands, the selected area was magnified and slow scan with a step size of 20 nm was conducted. The corresponding high-magnification Kikuchi pattern quality (KPQ) map (Fig. 4a) composed of numerous straight dark lines in a micron-scale band (indicated with red line). To identify details of the band, misorientation along the red line in Fig. 1a was measured and the results was plotted in Fig. 4b. It shows that whenever the scan passes the lines, there will be a 60° misorientation, indicating that these bands are composed of $\Sigma 3$ twins. Some smaller bands may be missed due to the limited spatial

resolution of EBSD [26]. When the strain was increased to 19.5%, the density of thin parallel bands was remarkably increased and they are with various width and observed in both $\langle 1\ 1\ 1 \rangle$ grains and those with certain deviation (Fig. 2e). These bands are also identified to be $\Sigma 3$ twins with 60° misorientation with their surroundings. The BSE image (Fig. 5) shows that the microstructures are full of parallel bands with $\sim 2\ \mu\text{m}$ spacing; and each band consists of numerous nano-twins with $\sim 10\ \text{nm}$ thickness. Another striking feature is that the grains that are dominated by twinning bands also exhibit a secondary twinning system. When the strain is further increased to 32.5%, more grains are dominated by twinning bands with much smaller spacing (Fig. 2g). Interaction between two twinning systems was also observed (Fig. 2g and Fig. 6). When the material was further strained to failure, twin density continuously increases and a colony-like morphology is formed (Fig. 2i). The colony consists of deformation twins with varying thicknesses and spacings. Only a few parts of the microstructure are free of twin bands. A majority of grains exhibit a secondary twinning system.

As the deformation temperature increases from 77 to 293 K, the appearance of twinning is postponed. Both high-magnification BSE images and EBSD cannot reveal the existence of deformation twin for samples tensile-deformed to an engineering strain lower than 32.5%. At 32.5% tensile strain, a few sparsely distributed twin bands were observed (Fig. 2h). When the material was continuously deformed to fracture, the twin volume fraction increases significantly with the observation of denser and thicker twin bundles generated by more than one twinning systems.

4. Discussion

The current investigations indicate that the plastic deformation of the current alloy is strongly mediated by nano-twinning. A schematic illustration of the twinning activity as a function of tensile strain is shown in Figure 7. At the early stage of deformation, dislocation accumulation along the grain boundaries nucleates nano-scaled primary twins at the boundaries once a critical stress is reached. The number of twins increases with strain while their individual thickness remains almost constant. Numerous twins will bundle together to form twin bands. Gradually, the thick twin bands will occupy almost all grain volume. When the tensile strain is further increased, more and more twins will bundle together and simultaneously or subsequently the secondary twinning systems will be activated. More and more grains will show more than one twinning systems. The twins generated from different systems will interact with each other, making a zig-zag-like microstructure.

The factors that have potential of affecting the above-mentioned twinning activities will definitely influence materials' mechanical performance (e.g., strain hardening). There is clear evidence from the current study that a few factors will affect the twinning activities, including alloying addition, grain orientation, and temperature. Thus, each of these will be discussed separately.

Carbon effects. In the base-alloy FeNiCoCrMn, at 77 K, nano-twinning was observed after ~ 20% strain and act as additional deformation mode. When the alloy was deformed beyond ~ 38% engineering strain, additional twinning systems were activated in the {1 1

1} twinning planes. No nano-twinning was observed for the FeNiCoCrMn alloy tensile-strained at 293 K [12]. Compared to these observations, when the material is alloyed with 0.5 at.% carbon, the activation of primary and secondary twinning systems occurs at much lower strains (6.5% and 19.5%, respectively) at 77 K. Extensive twinning is even observed when the material is deformed to fracture at 293 K. All of these observations suggest more pronounced twinning activities in the FeNiCoCrMn-0.5C alloy than that in the FeNiCoCrMn alloy. Because of the strong solid solution hardening of the carbon interstitial atom in HEAs, the dislocation mediated plasticity (slips) become much more difficult in the C-containing HEAs; thus, the critical stress needed to activate twinning can be easily achieved at relatively early stage of deformation. In addition, the intrinsic stacking fault energy has been known to be an essential parameter determining the tendency of twinning. Twinning will be more pronounced in materials with lower the stacking fault energy. Similar to the effects of C on the stacking fault energy of Fe-based materials (e.g., austenitic steel [27]), the current observations suggested that the stacking fault energy of the equiatomic FeNiCoCrMn alloy is decreased by the addition of C.

This enhanced twinning may explain the higher engineering strains needed to achieve the maximum tensile stress in the C-free FeNiCoCrMn equiatomic alloy compared to the 0.5 at%-FeNiCoCrMn alloy. Specifically, in C-free FeNiCoCrMn, the maximum tensile stresses were achieved at ~85% strain (77 K, 820 MPa) and ~65% strain (293 K, 520 MPa), compared to ~70% strain (77 K, 1090 MPa) and ~40% strain (293 K, 655 MPa) in the 0.5 at% C alloy.

A detailed explanation to the correlation between the twinning activity and strain hardening is given below. Annealing twins have their thickness of more than 10 μm ,

therefore their obstacles to dislocation movement is weak [28]. In contrast, the non-bundled thin mechanical twins (normally in a few to tens nm scales) formed with a low density (e.g., some individual twins observed in FeNiCoCrMn-0.5C alloy at 6.5% strain (Fig. 4a) will be able to serve as effective obstacles to dislocation motion. They can act as even stronger obstacles than single twins once they are arranged in bundle (e.g., >13%) due to the higher critical stress required to carry plastic deformation across the twin bundle than that required to penetrate an individual twin. Furthermore, interactions between dislocations and twins will cause the accumulation of a high density of sessile dislocations within the twin lamellae, leading to increased twin strength with deformation. These accumulated dislocations are potentially effective barriers to dislocation motion and can provide additional strengthening within the induced mechanical twins and increase the critical stress required to induce plastic deformation across the twins [29, 30]. A map revealing short-range orientation gradients were shown in Figure 8a, supporting the hypothesis of dislocation accumulation around the twin boundaries. The orientation gradients were highlighted using the Kernel parameter for the first neighbors which is the average misorientation angle between the current pixel orientation and its six first neighbors. The corresponding scanned area was marked in Fig. 2i by the arrow. The results show that the orientation variations exhibit higher values near twin boundaries. This could be caused by the twin-slip interactions and may indicate the dislocation pile-up at the twin boundaries. With increasing strain, the development of a dense twin substructure (Fig. 2g and 2i), the cutting of twin boundaries through dislocation substructure and the interactions between primary and secondary twinning systems will result in a further microstructure refinement. The effect is often referred to

as a “dynamic Hall-Petch effect” [31-36]. Figure 8b provides a further microstructural support of this effect. Upon failure at 77 K, their microstructures are separated by twin (lamellae) boundaries into numerous tiny twin-free areas due to the intensive interactions between twin bands from different twinning systems. Because the deformation-twin boundaries will be able to serve as dislocation-motion obstacles, each twin-surrounded area will act as a “grain”, making the “effective grain size” much smaller than the real grain size.

Grain orientation effects. A strong orientation-dependence of the twinning activities was observed. Under tension, twinning tends to be activated on the $\langle 1\ 1\ 1 \rangle$ //TA oriented grains and those with certain deviations. Similar observations have been reported for a few face-centered cubic metal and alloys, including Cu [37], Ag–Au alloys [38], and polycrystalline Fe-33Mn-3Al-3Si [39], Fe-31Mn-3Al-3Si [40], and Fe-22Mn-0.6C [41] TWIP steels. In all of these materials, deformation twinning readily occurred when the tensile axis lay near $[1\ 1\ 1]$ direction, whereas no deformation twinning was observed for the orientation lying near the $[0\ 0\ 1]$. These dependencies of the mechanical twinning activation on the grain orientation have been explained by assuming that the critical resolved shear stress for slip is identical to that for mechanical twinning [39, 41, 42]. In this case, grains with different orientations will have different preferred deformation mechanism and this could be evaluated by comparing the Schmid factors for dislocation slip and mechanical twinning. Under tension conditions, in grains with their $\langle 1\ 1\ 1 \rangle$ directions aligned to the tensile axis (TA), the Schmid factor for the mechanical twinning (0.31) is larger than that for dislocation slip (0.27); while in $\langle 0\ 0\ 1 \rangle$ //TA oriented grains the Schmid factor for dislocation slip (0.41) is larger than that for deformation twinning

(0.24) [42]. Another explanation is based on the assumption that the formation of mechanical twinning is governed by the dissociation of glide dislocations into Shockley partials only [43]. In this case, the degree of separation of two moving Shockley partials (leading and trailing partials) will be one of the most important factors determining the twin formation. It was suggested that the steady-state separation of a glide dislocation (width of partial separation) will show strong orientation dependence, and in $\langle 1\ 1\ 1 \rangle$ oriented grains, complete dissociation of the Shockley pair will occur with the production of stacking faults, but in $\langle 0\ 0\ 1 \rangle$ oriented grains, the dislocation pair will experience a constriction [44, 45]. Convincing microstructural evidence was provided by Idrissi [46] to show that these stacking faults can act as precursors for the formation of deformation-induced twins. Further study is needed to have a complete understanding of this.

Temperature effects. The temperature effects on twinning can also be clearly seen by comparing the microstructures of the investigated alloy at different temperatures. For the FeNiCoCrMn-0.5C alloy, while it was deformed at 293 K, the appearance of deformation twinning occurs at an engineering strain of 32.5% while twinning happens at very early stages of deformation (e.g., 6.5%) at 77 K. The activation of secondary twinning systems occurs also at a much earlier stage when the materials are strained at lower temperatures. The deformation temperature may influence the twinning ability through several different ways. First, it has been widely accepted that there exist a critical stress that needs to be reached in order to activate deformation twinning [47-50]. Under higher temperatures (e.g., 293 K), this stress will be more difficult to reach due to the lower yield strength or dynamic recovery. However, when the deformation temperature is lower (e.g., to 77 K), the dynamic recovery is reduced, and the yield strength will increase (Fig. 2 and Table 1).

The increased yield strength will make it easier to reach the critical stress at lower strain. Second, deformation temperature will affect twinning by changing the stacking fault energy of material. Stacking fault energy decrease at lower temperature and will cause more twinning.

5. Summary and Conclusions

The alloying design strategy of TWIP steels is successfully applied to a newly developed single-phase FCC high entropy alloy, FeNiCoCrMn, which has been shown to exhibit promising strength and ductility for cryogenic applications. To achieve this end, carbon is added to modify its microstructure (e.g., twinning activity) and thus mechanical properties (e.g., strain hardening). The microstructures and mechanical properties were characterized as a function of temperature, strain and carbon concentration. The results were compared with those of previous-reported on the base alloy-FeNiCoCrMn. Based on current results and analyses, the following conclusions could be drawn:

- (1) Adding 0.5 at.% carbon will not change the single-phase feature of equiatomic FeNiCoCrMn alloy during casting, homogenization, cold-rolling, annealing or further tensile-straining.
- (2) For 0.5 at.% C-containing FeNiCoCrMn alloy, both strength (yield strength and ultimate tensile strength) and ductility generally increase with decreasing temperature with more pronounced difference between 293 and 77 K.
- (3) Both yield strength and ultimate tensile strength were increased by the addition of 0.5 at.% carbon concentration while a reverse trend was observed for the uniform

elongation, indicating a negative dependence of average strain rate on carbon concentration.

(4) The increased strain hardening and strength could be caused by the promptness of deformation twinning in C-containing high entropy alloys. The twinning ability and activities were found to be affected by a few factors, including grain orientation, temperature and carbon concentration. Under current tensile condition, twinning is more pronounced in grains with their $\langle 111 \rangle$ directions aligned with the tensile axle; while in $\langle 001 \rangle$ oriented grains twinning rarely occurs. Twinning activities are enhanced by decreasing deformation temperature and increasing carbon addition. Further investigations are needed to have a thorough understanding of these dependencies.

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Tables:

Table I: 0.2% offset yield stress (σ_y), the ultimate tensile strength (UTS), the uniform elongation to fracture (e_f), extent of work hardening for the investigated alloy ($\Delta\sigma = \text{UTS} - \sigma_y$).

	FeNiCoCrMn [12]		FeNiCoCrMn-0.5C	
	77 K	293 K	77 K	293 K
σ_y (MPa)	350	165	510	225
UTS (MPa)	820	520	1090	655
e_f (%)	85	65	69	38
$\Delta\sigma$ (MPa)	470	355	590	430

Figure Captions:

- Fig. 1. (a) Microstructure of FeNiCoCrMn-0.5C alloy annealed at 1100 °C for 1 hour after cold-rolling; coloring is the inverse pole figure map projected on rolling direction (horizontal) (b) Engineering stress vs. plastic strain as a function of temperature for FeNiCoCrMn-0.5C alloys, for comparison, the stress-strain curve of the C-free FeNiCoCrMn with grain size of 155 μm [12] is also included.
- Fig. 2. EBSD images of FeNiCoCrMn-0.5C alloy shows the twinning evaluations as a function of strains at two different temperatures of (a, c, e, g, i) 293 K and (b, d, f, h, j) 77 K. Coloring is the inverse pole figure map projected on tensile direction (horizontal)
- Fig. 3. Back-scatter electronic image of FeNiCoCrMn-0.5C alloy deformed at 6.5% strain at 77 K.
- Fig. 4. (a) High-magnification Kikuchi pattern quality (KPQ) map of FeNiCoCrMn-0.5C alloy tensile-deformed at 13% engineering strain at 77 K; (b) corresponding misorientation as a function of distance for the red line.
- Fig. 5. Backscatter electron image of FeNiCoCrMn-0.5C alloy tensile-deformed at 19.5% engineering strain at 77 K.
- Fig. 6. Backscatter electron image of FeNiCoCrMn-0.5C alloy tensile-deformed at 32.5% engineering strain at 77 K.
- Fig. 7. Schematic illustration shows the evolution of twinning activities as a function of strain. For simplicity, the change of grain shape with deformation is not sketched.
- Fig. 8. (a) The kernel average misorientation map showing the degree of orientation gradients between the current pixel orientation and its six first-nearest neighbors, and (b) the combined index of image quality and inverse pole figure (projected onto tensile axis) map of the selected area arrow-marked in Figure 2i.

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