

Plasticity induced by nanoindentation in a CrCoNi medium-entropy alloy studied by accurate electron channeling contrast imaging revealing dislocation-low angle grain boundary interactions

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27 Abstract

In the present work, interactions of nanoindentation-induced dislocations (NIDs) 28 with a low-angle grain boundary (LAGB) are investigated in a single-crystalline 29 CrCoNi medium-entropy alloy (MEA). Microstructural evolutions before and after 30 31 nanoindentation were examined using accurate electron channeling contrast imaging. In the as-grown state, the alloy microstructure consists of subgrains 32 separated by LAGBs. After nanoindentation on the (001) plane far away from 33 LAGBs, the load-displacement curves exhibit the typical behavior of metals and 34 alloys with a pop-in marking the elastic-plastic transition. This pop-in is related to 35 the nucleation of NIDs that are observed to form pile-ups on {111} planes. In 36 contrast, when indents are performed in the vicinity of a LAGB with a low 37 misorientation angle of 0.24° and consisting of dislocations spaced ~60 nm apart, 38 different micromechanical responses and deformation mechanisms are observed 39 depending on the distance between the LAGB and the nanoindenter tip. When 40 the distance between the LAGB and the nanoindenter tip is larger than four times 41 42 the size of the indent (corresponding ratio: R > 4), the LAGB does not affect the micromechanical response nor interact with NIDs. In contrast, when the indenter 43 comes in direct or indirect contact with the LAGB (R < 1), the load-displacement 44 curve deviates at low loads from the elastic stage, and pop-ins are not observed. 45 In this case, the continuous deformation is accommodated by the movement of 46 the pre-existing LAGB dislocations. For intermediate cases with 1 < R < 4, the 47 load of the initial pop-in is dependent on the local defect density. In this latter 48 case, the pile-ups of NIDs directly impinge on the LAGB. Microstructural analyses 49 reveal that the LAGB accommodates plasticity by blocking the NIDs, activating a 50

51 dislocation nucleation site in the adjacent subgrain/emission of dislocation from

52 the LAGB, and inducing slight motions of its constituent dislocations.

53 Keywords: dislocations, low-angle grain boundary, NiCoCr medium-entropy alloy,

54 A-ECCI, nanoindentation.

55 1. Introduction

Medium-entropy alloys (MEAs) and high-entropy alloys (HEAs) consist of two to 56 four and at least five elements, respectively, with nearly equiatomic 57 concentrations [1-2]. Among these new classes of materials, the CrMnFeCoNi 58 HEA and one of its equiatomic ternary derivatives, the CrCoNi MEA, are currently 59 receiving increasing interest in the literature [3-4,5] because they both exhibit 60 outstanding damage tolerances at 77 K and 293 K with CrCoNi showing superior 61 properties. This alloy possesses an exceptional combination of tensile strength 62 (1.3 GPa), ductility (up to 90%), and fracture toughness (275 MPa·m^{-1/2}) at 77 K 63 64 [6], even superior to that of high-end cryogenic high Ni-steels [7]. This makes the equiatomic CrCoNi MEA a promising alloy for low-temperature applications [8]. 65

Some of the reasons for the outstanding damage tolerance of the CrCoNi MEA 66 are low stacking fault (SF) energy (22 ± 4 mJ·m⁻² [9]) and high shear modulus 67 [10] that promote strong work hardening rates (*e.g.*, forest hardening is directly 68 proportional to the shear modulus). The low SF energy does not only promote 69 planar glide of dissociated dislocations and hinders cross-slip, but also favors 70 deformation nanotwinning. This latter deformation mechanism introduces 71 coherent interfaces acting as barriers to dislocation motion and results in a 72 dynamic Hall-Petch effect without reducing ductility [6,9,11-12]. From a more 73

general point of view, the strengthening due to grain boundaries and annealing 74 twin boundaries (Hall-Petch strengthening) in the CrCoNi MEA was reported to 75 be particularly effective with a Hall-Petch slope of 600 MPa·m^{-1/2} [13]. However, 76 even though promising modeling advances were recently reported in the 77 literature [14-15], there is still a lack of understanding regarding the relative 78 contributions of different types of boundaries to the overall yield strength, *i.e.*, 79 interactions of dislocations with different types of boundaries, such as low-angle 80 grain boundaries (LAGB), coherent twin boundaries and general high-angle grain 81 82 boundaries. In the present study, we investigated LAGB-dislocation interactions in a single-crystalline CrCoNi MEA. This choice is motivated by recent studies of 83 HEAs and MEAs single crystals for which a significant scatter of critical resolved 84 shear stresses (CRSS) was reported in the literature [16-18]. As these alloys are 85 chemically complex and exhibit dendritic solidification, the LAGB density may 86 87 strongly vary from one study to another depending on the growth rate. As LAGBs resist the motion of dislocations, different LAGB densities are probably 88 responsible for the observed scatter of CRSS values [19]. Therefore, it is 89 important to characterize LAGB-dislocation interactions in MEAs and HEAs to 90 better understand how LAGBs contribute to the strength of MEA and HEA single 91 crystals. 92

From a more general viewpoint, much of what is known about dislocation-LAGB interactions in pure metals and alloys mainly comes from numerical studies by discrete dislocation dynamics (DDD) and molecular dynamics (MD) [20-24] and sometimes from theoretical studies [25] because experimental investigations are limited due to difficulties related to carrying them out. For example, Kappor and

Verdhan [23] studied the interactions of pile-up dislocations with low angle tilt 98 boundaries in face-centered cubic (FCC) crystals. The authors showed that the 99 interactions occur by collinear annihilation along with Lomer and glissile junction 100 formations [23-24]. Similarly, the formation of symmetric and asymmetric Hirth 101 Locks, as well as direct transmission of dislocations, were found to be the main 102 interactions between dislocations and Lomer-type LAGB in pure Ni using MD 103 simulation [25]. Only a handful of experimental investigations are available and 104 105 were mainly performed by *in-situ* and *ex-situ* tests inside a transmission electron 106 microscope (TEM). The impediment of motion and trapping of lattice dislocations by the LAGB as well as the emission of dislocations were observed during in-situ 107 108 nanoindentation inside the TEM in SrTiO₃ [26] and Fe-0.4wt%C [27]. The LAGBs were also found to act as barriers to dislocation motion and as nucleation sites in 109 martensitic steels during in-situ micromechanical testing in the TEM [28]. Further 110 experimental investigations of dislocations-LAGB interactions are important 111 because LAGBs are prevalent in single crystals of metallic materials [29], 112 113 annealed metals and alloys [30-33], ultrafine-grained materials [34-37], and 114 martensitic steels [28]. They also pave the way for the understanding of intergranular plasticity [38-39]. 115

From a fundamental point of view, the relationship between microstructure evolution and mechanical properties near GBs is not fully understood. Various attempts to bridge such experimental shortages were done by carrying out nanoindentation in the vicinity of GBs, analyzing the micromechanical responses, and theoretically linking them with dislocation activities without any dislocation scale imaging [40-43]. Soiffer et al. [40] found that hardness values increased by

122 33% in the vicinity of GBs compared to the hardness values inside grains in pure Cu and attributed this behavior to GB steps produced by electropolishing, the 123 anisotropy of dislocation-GB interactions, etc. Later on, the authors linked 124 dislocation transmission across GBs to the initial pop-in event on the load-125 displacement curve for indents performed between 0.2 < R < 0.4 (R is the ratio 126 of the distance to the GB to the residual indent size) in a BCC Fe-14wt% Si 127 because they did not observe pop-ins in the matrix (far away from the GBs) [41]. 128 In contrast to these observations, Wang and Ngan [42] observed two large pop-129 ins when nanoindentation tests were performed close to GBs in pure niobium. 130 They attributed the first and second pop-ins to incipient plasticity and the sudden 131 132 transmission of dislocations across the GB, respectively. A similar interpretation was also provided by Pöhl [43] who investigated the occurrence of pop-ins in a 133 pure iron polycrystal. However, the author also pointed out that several other 134 mechanisms (see Fig. 8 of Ref. [43]) and factors (surface roughness, initial 135 dislocation density...) could be responsible for strain bursts observed during 136 137 nanoindentation. Since there are several possibilities to account for the occurrence of strain bursts during nanoindentation, these tests must be 138 accompanied by dislocation-scale observations, which is one of the objectives of 139 the present study. In this context, instrumented nanoindentation is used to locally 140 induce plasticity in the vicinity of a LAGB. Then, accurate electron channeling 141 contrast imaging (A-ECCI) is employed post mortem to monitor the resulting 142 143 microstructural changes. A-ECCI is chosen here because it allows to image and characterize defects such as individual dislocations [44-46], LAGBs [47], SFs [48] 144 in relatively large areas at depths up to ~100 nm below the specimen surface. 145

Indeed, imaging of dislocation distribution is very crucial for understanding what happens in the vicinity of the LAGB after nanoindentation. For more details about the working principle of A-ECCI, the reader may refer to Refs. [44,49]. The combination of these two techniques, therefore, allows us to examine how nanoindentation-induced dislocations (NIDs) interact with LAGBs and link these interactions to micromechanical responses in an alloy with a low SF energy.

152 153

2. Materials and methods

An [001]-oriented single crystal of the $Cr_{33.3}Co_{33.3}Ni_{33.3}$ (at.%) MEA was processed by the seeded Bridgman technique using a CMSX-4 type Ni-base superalloy [10,50]. A cylindrical cross-section with a diameter of 12 mm and a height of 8 mm was cut at 90° from the growth axis. The sample was polished with diamond suspensions down to 1 µm and then subjected to chemomechanical polishing in an aqueous solution of hydrogen peroxide mixed with an oxide suspension to obtain a deformation-free surface.

Microstructural characterization was performed using A-ECCI and high-resolution 161 electron backscatter diffraction (HR-EBSD) in a Zeiss AURIGA SEM 162 (Oberkochen, Germany). Note that the sample was not repolished after 163 nanoindentation. The ECC micrographs were acquired using a standard 164 four-quadrant silicon diode backscattered electron (BSE) detector placed under 165 the pole piece using an acceleration voltage of 20 keV, and a 6.1 mm working 166 distance. EBSD maps were acquired with a commercial EBSD system, Aztec 167 (Oxford Instrument, Inc., UK) using the same acceleration voltage and a step size 168 of 0.35 µm. The sample was mounted on a holder pre-tilted at 70° to the 169

horizontal. Post-processing of the map was carried out using the HKL Channel 5
(Oxford Instrument, Inc., UK) and ATEX [51] (LEM3, France) software packages.

Compositional analyses were carried out using energy-dispersive X-ray spectroscopy (EDX) in an SEM of type JEOL JSM-IT 300 equipped with an Octane Elite EDAX Ametek detector and the analytical software EDAX Genesis (v.6.53). An accelerating voltage of 25 kV in combination with a probe current of 60 mA, a working distance of ~10 mm, and a count rate of ~150.000 counts/s were used. A total number of 1024 frames were collected, resulting in a total recording time of ~10 h.

Local plastic deformation was introduced in the vicinity of a LAGB using a nanoindenter (Hysitron TI-900, Bruker, US) with a Berkovich tip (Synton-MDP AG, Switzerland). The latter is chosen because it is sharp with a small tip radius of less than 200 nm, suitable for placing indents as close as possible to LAGBs [52]. Nanoindents were performed using a load-controlled mode and peak load of 500 µN. They were spaced at least 3 µm apart to avoid any mutual interaction.

185 3. Results

186 3.1. Microstructure before nanoindentation

187 3.1.1. Microstructure overview of the single crystal

The equiatomic CrCoNi MEA crystallizes with a single-phase FCC structure [9]. A representative BSE micrograph (Fig. 1a) of the sample showcases various randomly oriented dendrites with variable sizes (average: \sim 100 µm) and contrasts. Such a dendritic morphology is also referred to as crystal mosaicity in the literature and was, for example, observed in Ni-base superalloy single crystalsgrown by the same technique [50,53].

An HR-EBSD map was acquired for the area marked with a white frame in Fig. 1a 194 195 for detailed investigation. The corresponding misorientation map in Fig. 1b reveals that the misorientation angles (θ) between neighboring dendrites can be 196 as small as 0.2° and that most of them are lower than 2°. Therefore, these 197 dendrites are separated by LAGBs since the misorientations are lower than 10° 198 [26]. These dendrites are also referred to as subgrains. To obtain a precise 199 orientation of the subgrain marked with a white frame in Fig. 1b, the procedure 200 201 reported in Ref. [44] was followed, *i.e.*, a high-resolution selected area channeling 202 patterns (e.g, white frame in Fig. 1c) were recorded at different tilt and rotation 203 angles and superimposed on a simulated Kikuchi pattern for indexing. From the 204 area of the subgrain highlighted with a white frame in Fig. 1b, the ECC micrograph shown in Fig. 1d was acquired with the diffraction vector $g = (2\overline{2}0)$. 205 Figure 1d shows asymmetrical wing-shaped spots with a black and white (B-W) 206 contrast. Each spot corresponds to a trace on the surface of a threading 207 dislocation (TD), *i.e.*, a dislocation nearly perpendicular to the surface. Moreover, 208 there is a highly inclined dislocation that appears with a comet-like contrast (white 209 210 circle in Fig. 1d). The same area was imaged with g = (220) and a similar number of dislocations were observed. 211



212

0.6* 0.15 0.2* 0.3*

Fig. 1. (a) BSE micrograph of an [001]-oriented single-crystal of the CrCoNi alloy. 213 214 (b) Grain boundary misorientation map of the white-framed region in (a). (c) Highresolution selected area channeling pattern (HR-SACP) from the white frame in (b) 215 superimposed onto a simulated Kikuchi pattern where the red cross marks the optical 216 217 axis of the SEM. (d) ECC micrograph taken inside the subgrain highlighted with a white frame in (b) using $g = (2\overline{2}0)$, see white arrow in (c). (e) Grain boundary misorientation 218 map of the black-framed region in (b) superimposed onto a subgrain orientation map. 219 220 Here, ECC analysis was performed at the region framed in white. (f) ECC micrograph of 221 the LAGB under $g = (4\overline{2}0)$. Note that the HR-SACP shown in the upper right corner of (f) 222 is not superimposed onto the simulated Kikuchi pattern in (c) for readability reasons.

223 The TDs offer an advantage over curved or straight dislocations [54] because they allow a better estimation of the dislocation density (ρ) using Eq. (1): 224

$$\rho = \frac{N}{A} \tag{1},$$

where *N* is the number of TDs and *A* is the surface area of the micrograph [47]. The ρ value averaged over 20 micrographs (total area: 140 μ m²) taken inside five different subgrains is (3.6 ± 0.9) × 10¹² m⁻². This value is comparable to that found in as-cast single crystals [55].

Figure. 2a shows a BSE micrograph where several subgrains/dendrites can be 230 observed. The EDX maps shown in Figs. 2b-d display the distribution of chemical 231 232 elements within the dendritic microstructure and the results of EDX point analyses, performed at locations highlighted by the numbers 1-4 in interdendritic 233 and 5-8 in dendritic regions, are summarised in Table 1, respectively. The 234 235 dendrites are enriched in Co and depleted in Cr with an average composition of $Cr_{30}Co_{36}Ni_{34}$ and have a size of ~100 μ m. In contrast, interdendritic regions are 236 ~25 µm wide with an average composition of Cr₃₅Co₃₂Ni₃₃. 237



239

Fig. 2. (a) BSE micrograph of an [001]-oriented single crystal of the CrCoNi alloy. EDS
maps of (b) Cr, (c) Co, and (d) Ni.

Table 1. Chemical composition in dendritic and interdendritic regions (in at.%) for

243 positions in Fig. 2c and 2d

Interdendritic regions				Dendritic regions			
N°	Cr	Co	Ni	N°	Cr	Со	Ni
1	35.0	31.5	33.5	5	30.0	36.0	34.0
2	35.0	32.0	33.0	6	30.5	35.5	34.0
3	35.0	31.5	33.5	7	30.0	36.0	34.0
4	35.0	31.5	33.5	8	30.0	35.5	34.0

Since all our nanoindents were performed at distances that are at most 12 μ m away from the LAGB (see Fig. 3a), the mechanical responses are therefore representative of the composition present in the interdendritic regions (Cr₃₅Co₃₂Ni₃₃).

249 3.1.2. Structure of the LAGB of interest

The region marked with a black frame in Fig. 1b is magnified in the crystallite orientation map shown in Fig. 1e where the red color indicates that the crystallites have near {001} orientations. Figure 1e is centered on a LAGB of interest that separates subgrains A and B. The range of θ -values along the LAGB is 0.15°-0.3° as deduced from the misorientation map. Assuming that this LAGB consists of an array of equally spaced dislocations, invoking the model of Read and Shockley [55], the expected spacing between dislocations (*d*) can be estimated using:

257
$$d = \frac{|\mathbf{b}|}{2\sin(\theta/2)}$$
 (2),

where **b** = $\frac{1}{2}$ a <110>. Using θ = 0.15-0.3° in combination with Eq. 2, we find d values between 100 and 50 nm. The ECC micrograph (Fig. 1f) acquired with **g** = ($4\overline{2}0$) in the white-framed region of Fig. 1e shows that the LAGB consists of an array of TDs with a mean spacing d = 60 ± 5 nm (see the yellow bracket in Fig. 1f). From this value, an average misorientation of 0.24 ± 0.02° can be deduced from Eq. 2. This latter value is consistent with the misorientation angle determined by EBSD and validates our approach using A-ECCI.

Due to the surface relaxation effect, the Burgers vectors of the TDs within grains in Fig. 1d and along the LAGB in Fig. 1f cannot be determined using the invisibility criteria $\mathbf{g} \cdot \mathbf{b} = 0$ and $\mathbf{g} \cdot \mathbf{b} \times \mathbf{u} = 0$, where \mathbf{u} is a unit vector along the dislocation line [44-45,56-57] because these dislocations exhibit a residual contrast even if the invisibility criteria are fulfilled. Therefore, it is not straightforward to determine whether a particular dislocation has an edge, mixed, or screw character. However, Tunstall and Hirsch [56] showed by theoretical calculations and TEM imaging in Pt and Cu that the B-W contrast of TDs is stronger and laterally larger for screw TDs than edge TDs. As the TDs along the LAGB (Fig. 1f) show varying contrasts, this implies that the boundary has a mixed tilt and twist character.

275 3.2. Mechanical response and microstructure after

276

nanoindentation

An array of 56 indents was performed in the vicinity of the LAGB of interest. 277 Among these indents, 15 indents, surrounded by a red line in Fig. 3a, were 278 279 selected and the load at which the first pop-in was observed on the corresponding load-displacement curves (to be described in Section 3.2.1) was plotted against 280 the distance to the LAGB in Fig. 3b (see x-axis at the top) and the normalized 281 distance R (ratio between the distance to the LAGB and the indent size, see 282 bottom x-axis in Fig. 3b). Since the indent size is ~0.5 μ m, R is twice the distance 283 284 to the LAGB. This plot shows that the distribution of pop-in loads is centered around a mean value of 158 μ N with a standard deviation of 23 μ N when R > 1285 286 (black dashed line in Fig. 3b). This distribution can be attributed to the initial local defect density prior to nanoindentation [43]. It also shows that the indents closest 287 to (R < 1, #4 and #3 in Fig. 3a) and in contact with the LAGB (#5) exhibit a pop-in 288 at the elastic-plastic transition that is either significantly lower than the mean 289 290 value (#3) or simply absent (#4 and #5). In addition, two indents (#1 and #2 in Fig. 3a) inside subgrains A and B were investigated. 291



Fig. 3. (a) Array of 7×8 indents around the LAGB. The indents surrounded by the red line and that are close to the LAGB were used to plot the load of the initial pop-in against distance to the LAGB in (b). These distances were also normalized with respect to the size of the remnant indent, see ratio *R* at the bottom *x*-axis of (b). Numbers and colored circles show indents that are analyzed in detail in Section 3.2. The black dashed line in (b) shows the average initial pop-in load of indents when R > 1.

These indents were chosen because they show similar low initial dislocation densities and they are far enough from the LAGB such that their local micromechanical responses are not affected. The next sections compare the microstructure evolution and mechanical response of the five above-mentioned indents and the results are presented in Figs. 4-7.

304 3.2.1. Nanoindentation inside subgrains

We now present the micromechanical responses and microstructural evolutions from the representative indents performed in the interior of the subgrains. Nanoindentation tests were performed in subgrains A and B (indents #1 and #2 shown in Figs. 3 and 4a) at locations with a low initial dislocation density

 $(\rho = 2.02 \times 10^{12} \text{ m}^{-2})$. Their load-displacement (*P*-*h*) curves are shown for 309 subgrains A and B as black and red curves in Fig. 4b, respectively. They exhibit 310 311 an initially purely elastic loading that can be described by the Hertzian contact theory, see the dotted light blue curve in Fig. 4b. This stage is followed by a 312 sudden deformation event also known as initial pop-in (black arrow in Fig. 4b) at 313 critical loads of 194 µN and 185 µN in the subgrains A and B, respectively. While 314 initial pop-ins have been linked with different mechanisms [58], it is generally 315 316 associated with the elastic-plastic transition, *i.e.*, the homogeneous nucleation of dislocations, which precedes their multiplication and motion to form pile-ups [59]. 317 On the red load-displacement curve in Fig. 4b, this initial pop-in is followed by 318 319 multiple small yield excursions highlighted by red arrows. These excursions, also 320 known as staircase yielding, are associated with the unlocking of pre-existing pinned dislocations [47] or displacement of dislocations produced under the 321 indenter [46,60-61]. 322

Figures 4c-d display micrographs of the local microstructures before and after 323 324 nanoindentation in subgrain A. Pre-existing threading dislocations (PEDs) are shown in Fig. 4c. Pile-ups of NIDs (blue arrow in Fig. 4d) are observed along the 325 traces of the {111} planes. These observations are consistent with the fact that 326 327 plastic deformation in the CrCoNi MEA was reported to occur by the planar glide of 1/2<110> dislocations dissociated into 1/6<112> Shockley partials on {111} 328 planes [9]. The bright areas, enclosed by the red ellipses in Fig. 4 and 329 330 subsequently in Figs. 6 and 7, corresponds to stacking faults SFs or nano-twins, which were also observed by ECCI in other materials [62-63], and were 331 theoretically calculated by Kriaa et al.[64]. 332

To differentiate PEDs and NIDs, a three-step procedure was followed. The ECC 333 micrographs before and after nanoindentation were acquired using the same 334 diffraction condition. Then, the two micrographs were superimposed to track the 335 positions of the PEDs (see GIF files in supplementary materials). Finally, new 336 dislocations that appeared after nanoindentation and formed pile-ups on {111} 337 planes were identified as NIDs. A schematic drawing of the deformed 338 microstructure in Fig. 4d is represented in Fig. 4e, where PEDs, NIDs and SFs 339 are marked in blue, yellow and red, respectively. As discussed in Section 3.1.1., 340 the Burgers vector of threading dislocations cannot be determined using 341 extinction conditions. Consequently, the symbols \perp used in Figs. 4e and h and 342 subsequently in Figs. 5-7 are used to mark the positions of dislocations and do 343 not refer to their type (since there is no standard symbol for dislocations with the 344 345 mixed character). The PEDs remained unaltered, probably because these dislocations lie outside the deformed region and do not interact with the new 346 NIDs. The pile-ups of the NIDs are visible on two sides of the indent on the traces 347 of the $(111)/(11\overline{1})$ and $(1\overline{1}1)/(1\overline{1}\overline{1})$ planes (yellow dislocations in Fig. 4e) and 348 their lengths (defined here as the distance between the outermost pile-up 349 350 dislocations and the center of the remnant indent) are ~0.68 µm and ~0.63 µm, 351 respectively.

A similar microstructural evolution was observed when nanoindentation was performed in subgrain B (Figs. 4f-h). However, the average length of the pile-ups on the (111)/(111) planes was longer (0.73 µm) than that in subgrain A. In contrast, the mean length of the pile-ups on the (111)/(111) planes was shorter (0.55 µm) and these pile-ups are located on the opposite side of the indent-



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Fig. 4. (a) Positions of the indents in the subgrains A and B superimposed onto the EBSD map (indents #1 and #2 in Fig. 3a). (b) Load-displacement curves in subgrains A and B. The initial elastic loading stage follows the Hertz contact theory. (c-h) Microstructural evolution as a result of nanoindentation in regions with a low initial local dislocation density ($\rho = 2.02 \times 10^{12} \text{ m}^{-2}$). (c-d) are ECC micrographs before and after nanoindentation in the subgrain A, respectively. (e) is a schematic of (d). (f-g) are ECC micrographs before and after deformation within subgrain B. (h) is a schematic of (g). The white dashed lines in (e and h) show the traces of the $(111)/(11\overline{1})$ and $(1\overline{1}1)/(1\overline{1}\overline{1})$ planes. The red ellipses in (d) and (g) and red thick lines in (h) and (e) mark the traces of SFs. NI and NID stand for nanoindentation and nanoindentation induced dislocation, respectively. Blue arrows in (d) and (g) show pile-ups directions. Note that the symbol \bot is used to mark the positions of dislocations and does not refer to their character.

-compared to subgrain A, *i.e.*, top left corner in Fig. 4d for subgrain A and bottom
right corner in Fig. 4g for subgrain B. The reasons for this difference may be
related to the small misorientation between subgrains A and B (compare Figs. 4c
and 4f).

375 3.2.2. Nanoindentation on and near the LAGB

When nanoindentation is carried out on the LAGB (Fig. 5a), its corresponding 376 load-displacement curve deviates early on from the Hertzian fit and does not 377 show any discernable pop-in (see the black curve in Fig. 6b). This 378 micromechanical response probably reflects the fact that, compared to 379 380 homogeneous nucleation of dislocations, much lower stresses are required to induce gliding of PEDs [43]. This interpretation is supported by the microstructure 381 that can be observed by A-ECCI around the residual indent, see Figs. 5c-d. 382 Indeed, the NIDs around the indent (yellow dislocations in Fig. 5d) are randomly 383 distributed and only one short dislocation pile-up is observed on one edge of the 384 indent. This microstructural observation together with the absence of pop-ins 385 suggests that the plasticity mostly occurred by the motion and multiplication of 386 pre-existing LAGB dislocations. 387





To investigate the interactions of dislocations with the LAGB, nanoindentation tests were performed at ~0.42 μ m (*R* = 0.84) from the LAGB in the subgrain A

 $(\rho = 4.02 \times 10^{12} \text{ m}^{-2})$ and at ~0.44 µm (R = 0.88) from the LAGB in the 398 subgrain B ($\rho = 4.08 \times 10^{12} \text{ m}^{-2}$), see Fig. 5a. Here, it is worth recalling that, far 399 away from the boundary, the pile-ups on the $(1\overline{1}1)/(1\overline{1}\overline{1})$ planes in subgrains A 400 and B were extending over 0.63 µm (Fig. 4d) and 0.55 µm (Fig. 4g), respectively. 401 We thus expect that nanoindentation tests #3 and #4 at distances lower than 402 0.5 μ m (R < 1) from the LAGB and displayed in Figs. 3a and 5a will result in the 403 formation of NID pile-ups that significantly interact with the LAGB and that the 404 micromechanical responses will be affected. Interestingly, 405 the red load-displacement curve (Fig. 5b), corresponding to the nanoindent performed in 406 407 the subgrain A at R = 0.84, does not show an initial pop-in and behaves mechanically like the indent that was performed on the LAGB. In contrast, the 408 green load-displacement curve in Fig. 5b, for the indent in the subgrain B at 409 R = 0.88, exhibits an initial pop-in at 132 µN which is significantly lower than the 410 mean value in this study. 411

412 The micrographs before and after nanoindentation in the subgrain A at R = 0.84are presented in Figs. 6a-d. Note that the resolution in Fig. 6b is higher than in 413 414 Fig. 6a due to the following reason. Since it is extremely difficult to precisely 415 indent at a specific location, a large area along and on each side of the LAGB $(25 \times 30 \,\mu\text{m}^2)$ was imaged with a reasonable resolution. Such imaging is done 416 417 to reduce acquisition time and avoid severe contamination of the area of interest by the electron beam *i.e.*, one has to find a compromise between acquisition time 418 419 and resolution. After nanoindentation, smaller areas can be imaged again with an enhanced resolution to better visualize the deformed microstructure. The 420

421 arrangement of the dislocation pile-ups and the presence of SFs were similar to the microstructures observed for indents performed far away from the LAGB 422 (compare Figs. 6b and 4d). The lengths of the pile-ups (yellow NIDs in Fig. 6d) 423 are ~0.57 µm on both traces of the {111} planes and are 10-16% shorter than 424 425 those for indents performed within the subgrains (far away from the LAGB). A local change of the LAGB structure is observed between LAGB dislocations 1 426 and 3 (dashed blue arrows in Figs. 6a-d) adjacent to the residual indent. Before 427 nanoindentation, the LAGB dislocations 1 and 2 are clearly aligned along the 428 green line shown in Fig. 6c. After nanoindentation, the LAGB dislocation 1 is now 429 430 at the end of a nanoindentation-induced SF while the LAGB dislocation 2 shifted slightly to the right along with other LAGB dislocations between 2 and 3 as 431 illustrated in Fig. 6d. For easier visualization of the slight motion of the LAGB 432 433 dislocations near the indent, an animated superimposition of the micrographs (Figs. 6a,b) before and after nanoindentation is provided as a GIF file in Fig. S1 434 (Supplementary data) where two dislocations located in subgrain A far away from 435 the indent (in the top left and top right corners of Figs. 6a,b) were used as markers 436 for the animation. To summarize, the LAGB accommodates plastic deformation 437 438 by the glide of its dislocations and this observation is consistent with the fact that no pop-ins were observed on the load-displacement curve (red in Fig. 5b). Even 439 though the pile-ups of NIDs were not visible on the side adjacent to the LAGB, a 440 new curved dislocation is found between the LAGB and the indent (see yellow 441 line in Fig. 6d). This dislocation may have been stopped by the LAGB or dragged 442 by the LAGB during its motion. 443



445 Fig. 6. (a-b) ECC micrographs before and after nanoindentation showing the evolution 446 of the dislocation configurations in the subgrain A at 0.42 μ m (*R* = 0.82) from the LAGB (indent #4 in Fig. 3a). (c-d) schematics of (a-b). The red ellipsis in (b) and red line in (d) 447 show an SF. The blue arrows in (b) show directions along which the NID pile-ups extend. 448 449 The blue dashed lines in (c) and (d) show the trace of the LAGB before and after 450 deformation. The green straight lines allow to compare the positions of the LAGB dislocations 1, 2, and 3 before and after nanoindentation while the green arrows show 451 the direction of the slight motion of LAGB dislocations. Note that the symbol \perp is used to 452 453 mark the positions of dislocations and does not refer to their character.

454 Similarly, Figs. 7a-b show the microstructural evolution before and after nanoindentation for the indent performed in the subgrain B at R = 0.88. It displays 455 pile-ups of NIDs (blue arrows in Fig. 7b) and two SFs (red ellipses in Fig. 7b) on 456 {111} planes. The lengths of the pile-ups of NIDs are 0.63 µm and 0.57 µm on 457 the traces of $(111)/(11\overline{1})$ and $(1\overline{1}1)/(1\overline{1}\overline{1})$ planes, respectively. Those on 458 $(1\overline{1}1)/(1\overline{1}\overline{1})$ were expected to interact with the LAGB and a change of the 459 dislocation structure of the LAGB was indeed observed as a result of the 460 nanoindentation test. To better observe these evolutions, high-resolution ECC 461 micrographs were recorded before and after nanoindentation at the location 462 marked with a blue frame in Figs. 7a-b. These micrographs are displayed in 463 464 Figs. 7c-d and their corresponding schematics showing the positions of individual 465 dislocations are displayed in Figs. 7e-f, where dislocations of interest are numbered from one to nine. As mentioned before, please note that the resolution 466 of Figs. 7b,d is higher than that used for Figs. 7a,c. After nanoindentation, NIDs, 467 shown as yellow dislocations in Fig. 7f, can be observed at ~40 nm from the 468 LAGB. These NIDs caused the rearrangement and the collective motion of the 469 pre-existing LAGB dislocations. The rearrangement is mainly visible between the 470 LAGB dislocations 5 and 8. Here, the dislocations form a ">" before 471 nanoindentation that expanded after nanoindentation (compare ellipses in 472 Figs. 7c and 7d). 473



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Fig. 7. Interactions between NIDs and the dislocations constituting the LAGB. (a-b) ECC micrographs before and after nanoindentation, respectively (indent #3 in Fig. 3). (c-d) Enlargement ECC micrograph of a portion of the LAGB before and after nanoindentation and (e-f) are their corresponding schematics. The red ellipses highlight SFs. Blue ellipses show dislocations that form a ">"-like shape. Note that the symbol \perp is used to mark the positions of dislocations and does not refer to their character.

The local collective motion of the LAGB dislocations between 1 and 9 away from the residual indent (toward the subgrain A) is shown in the animated superimposition of micrographs before and after nanoindentation, respectively, in Fig. S2 (supplementary data). Note that the two dislocations located in subgrain
A on the left of Figs. 7a,b were used as markers for this GIF file.

Furthermore, a new NID (highlighted by a red arrow in Fig. 7d) was observed in 486 subgrain A (compare Figs. 7c-d and 6e-f). Since the number of dislocations 487 remained constant in the boundary plane of the LAGB, mechanisms of 488 decomposition, transmission, absorption, combination, or reflection of NID are 489 490 not responsible for the above-mentioned dislocation generation [65-67]. Based on the position of the indents at ~0.45 µm from the LAGB and the expected pile-491 492 up size of >0.5 µm, it seems that the stress at the leading NID of the pile-up was high enough to activate a Frank-Read source in the subgrain A (note that this 493 source cannot be resolved because it is probably smaller than the resolution limit 494 of the A-ECCI method) or its position is deeper than the 100 nm that can be 495 imaged by A-ECCI. Alternatively, the new dislocation could have been emitted 496 from the LAGB. It is worth mentioning that the low misorientation angle (~0.24°) 497 between the two subgrains ensured that the geometrical criteria for dislocation 498 emission were satisfied [65-67]. 499

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4. Discussion

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502 By comparing the load-displacement curves, initial pop-in loads, and dislocation 503 microstructures for indents performed inside subgrains, near a LAGB, and on the 504 LAGB, it is found that the studied LAGB affects the local micromechanical 505 response and the underlying fundamental deformation mechanisms. Generally, 506 the micromechanical responses underneath an indenter in most situations are 507 dependent on the local microstructure such as the density of defects, their

508 configuration, and the local chemical composition [47]. At the LAGB, which consists of a special configuration of dislocations, the onset of plastic deformation 509 is initiated upon contact by the motion of its dislocations because there is no need 510 to nucleate new ones. This explains the absence of an initial pop-in on the load-511 displacement curve of indent #5 in Fig. 3. In contrast, nucleation of new 512 dislocations is needed to initiate plasticity far away from the LAGB and the 513 elastic/plastic transition on the load-displacement curve is thus marked by a 514 pronounced pop-in. 515

For the two indents at R < 1 in both subgrains A and B, their micromechanical 516 responses are extremely different despite being approximately at the same 517 518 distance from the boundary and having been placed in areas with similar dislocation densities prior to nanoindentation. Such discrepancy may have been 519 520 caused by three reasons. First, the local organization (discrete nature, character, and position of dislocations) and curvature of the LAGB are different for indents 521 #3 and #4, see Fig. 3a. Second, both indents are not symmetrical relative to the 522 LAGB plane, *i.e.*, the Berkovich indenter tip was not rotated around its axis 523 between the indents. Third, deformation mechanisms are also different as 524 evidenced by the different microstructures around the residual indents 525 (Section 3.2.2) 526

Interestingly the indents #5 and #4 performed on the LAGB and at R = 0.84 in subgrain A, respectively, do no exhibit pop-ins (see Fig. 3) and deviate at low loads from the Hertz law, even though they show different microstructure evolutions during nanoindentation. For indent #4 at R = 0.84, the absence of pop-in may be related to the sub-surface structure of the LAGB dislocations.

532 Indeed, if the LAGB is inclined towards the nanoindentation axis below the sample surface and deeper than the extinction depth of ECCI (~100nm), the 533 stress field below the indenter may have interacted with the inclined dislocations 534 of the LAGB, and plastic deformation was accommodated by the motion and 535 536 multiplication of these pre-existing LAGB dislocations.

The dendritic microstructure of single crystals resulting from different processing 537 538 routes and parameters was proposed to be one of the factors that may be at the origin of the significant scatter observed for the macro-mechanical CRSS of HEAs 539 and MEAs from the Cr-Mn-Fe-Co-Ni system [18-19]. From our study, it is found 540 541 that even a LAGB with a misorientation as low as 0.24° impedes the motion of 542 dislocations, modifies the local micromechanical response and the associated deformation mechanisms. Therefore, crystal mosaicity, related to the presence 543 544 of heterogeneously distributed LAGBs, which induce various misorientation angles within single crystals, is likely one of the factors responsible for the 545 scattering of macro-mechanical CRSS values reported in the literature. 546

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5. Conclusions

549 The following conclusions can be drawn from our findings. The LAGB of interest has an average misorientation of 0.24° and consists of dislocations aligned along 550 the [001] direction with a mean spacing of ~60 nm. Besides, the elemental 551 composition in the vicinity of this LAGB is Cr₃₅Co₃₂Ni₃₃ (in at.%) in a region that 552 is ~25 μ m wide while the subgrains (size: ~100 μ m) have an average composition 553 554 of $Cr_{30}Co_{36}Ni_{34}$. When nanoindentation tests are performed within subgrains with a maximum load of 500 µN, pile-ups of NIDs form on {111} planes around the 555

556 indents, and these pile-ups extend at least over ~0.45 µm. Therefore, when nanoindentation tests are performed at $\leq 0.5 \ \mu m \ (R \leq 1)$ away from the LAGB, it 557 is expected that the heads of the dislocation pile-ups interact with the LAGB. In 558 one case, the LAGBs blocked and absorbed the NID. In the other case, the LAGB 559 560 accommodated plastic deformation through the motion and local rearrangements of its dislocations, and a new dislocation was observed after deformation on the 561 other side of the LAGB. In terms of micromechanical response, an indent 562 performed on the LAGB does not show an initial pop-in as the pre-existing LAGB 563 dislocations control plasticity. Far away from the LAGB, the onset of plasticity on 564 the load-displacement curves was marked by pop-ins. The LAGB indeed affects 565 the local micromechanical response and fundamental deformation mechanisms 566 as evidenced by pop-ins and microstructural evolutions. The results of this work 567 bring new insights into dislocation/LAGB interactions and their respective 568 micromechanical responses in metallic materials, specifically, medium- and high-569 entropy alloys. 570

571 Declaration of Competing Interest

572 The authors declare that they have no known competing financial interests or 573 personal relationships that could have appeared to influence the work reported in 574 this paper.

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