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

5  
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## 27 Abstract

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

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

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

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

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

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

98 Verdhan [23] studied the interactions of pile-up dislocations with low angle tilt  
99 boundaries in face-centered cubic (FCC) crystals. The authors showed that the  
100 interactions occur by collinear annihilation along with Lomer and glissile junction  
101 formations [23-24]. Similarly, the formation of symmetric and asymmetric Hirth  
102 Locks, as well as direct transmission of dislocations, were found to be the main  
103 interactions between dislocations and Lomer-type LAGB in pure Ni using MD  
104 simulation [25]. Only a handful of experimental investigations are available and  
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  
107 by the LAGB as well as the emission of dislocations were observed during *in-situ*  
108 nanoindentation inside the TEM in SrTiO<sub>3</sub> [26] and Fe-0.4wt%C [27]. The LAGBs  
109 were also found to act as barriers to dislocation motion and as nucleation sites in  
110 martensitic steels during *in-situ* micromechanical testing in the TEM [28]. Further  
111 experimental investigations of dislocations-LAGB interactions are important  
112 because LAGBs are prevalent in single crystals of metallic materials [29],  
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  
115 intergranular plasticity [38-39].

116 From a fundamental point of view, the relationship between microstructure  
117 evolution and mechanical properties near GBs is not fully understood. Various  
118 attempts to bridge such experimental shortages were done by carrying out  
119 nanoindentation in the vicinity of GBs, analyzing the micromechanical responses,  
120 and theoretically linking them with dislocation activities without any dislocation  
121 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  
123 Cu and attributed this behavior to GB steps produced by electropolishing, the  
124 anisotropy of dislocation-GB interactions, etc. Later on, the authors linked  
125 dislocation transmission across GBs to the initial pop-in event on the load-  
126 displacement curve for indents performed between  $0.2 < R < 0.4$  ( $R$  is the ratio  
127 of the distance to the GB to the residual indent size) in a BCC Fe-14wt% Si  
128 because they did not observe pop-ins in the matrix (far away from the GBs) [41].  
129 In contrast to these observations, Wang and Ngan [42] observed two large pop-  
130 ins when nanoindentation tests were performed close to GBs in pure niobium.  
131 They attributed the first and second pop-ins to incipient plasticity and the sudden  
132 transmission of dislocations across the GB, respectively. A similar interpretation  
133 was also provided by Pöhl [43] who investigated the occurrence of pop-ins in a  
134 pure iron polycrystal. However, the author also pointed out that several other  
135 mechanisms (see Fig. 8 of Ref. [43]) and factors (surface roughness, initial  
136 dislocation density...) could be responsible for strain bursts observed during  
137 nanoindentation. Since there are several possibilities to account for the  
138 occurrence of strain bursts during nanoindentation, these tests must be  
139 accompanied by dislocation-scale observations, which is one of the objectives of  
140 the present study. In this context, instrumented nanoindentation is used to locally  
141 induce plasticity in the vicinity of a LAGB. Then, accurate electron channeling  
142 contrast imaging (A-ECCI) is employed *post mortem* to monitor the resulting  
143 microstructural changes. A-ECCI is chosen here because it allows to image and  
144 characterize defects such as individual dislocations [44-46], LAGBs [47], SFs [48]  
145 in relatively large areas at depths up to ~100 nm below the specimen surface.

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

## 152 2. Materials and methods

153

154 An [001]-oriented single crystal of the  $\text{Cr}_{33.3}\text{Co}_{33.3}\text{Ni}_{33.3}$  (at.%) MEA was  
155 processed by the seeded Bridgman technique using a CMSX-4 type Ni-base  
156 superalloy [10,50]. A cylindrical cross-section with a diameter of 12 mm and a  
157 height of 8 mm was cut at  $90^\circ$  from the growth axis. The sample was polished  
158 with diamond suspensions down to  $1\ \mu\text{m}$  and then subjected to chemo-  
159 mechanical polishing in an aqueous solution of hydrogen peroxide mixed with an  
160 oxide suspension to obtain a deformation-free surface.

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



170 horizontal. Post-processing of the map was carried out using the HKL Channel 5  
171 (Oxford Instrument, Inc., UK) and ATEX [51] (LEM3, France) software packages.  
172 Compositional analyses were carried out using energy-dispersive X-ray  
173 spectroscopy (EDX) in an SEM of type JEOL JSM-IT 300 equipped with an  
174 Octane Elite EDAX Ametek detector and the analytical software EDAX Genesis  
175 (v.6.53). An accelerating voltage of 25 kV in combination with a probe current of  
176 60 mA, a working distance of ~10 mm, and a count rate of ~150.000 counts/s  
177 were used. A total number of 1024 frames were collected, resulting in a total  
178 recording time of ~10 h.

179 Local plastic deformation was introduced in the vicinity of a LAGB using a  
180 nanoindenter (Hysitron TI-900, Bruker, US) with a Berkovich tip (Synton-MDP  
181 AG, Switzerland). The latter is chosen because it is sharp with a small tip radius  
182 of less than 200 nm, suitable for placing indents as close as possible to LAGBs  
183 [52]. Nanoindents were performed using a load-controlled mode and peak load  
184 of 500  $\mu\text{N}$ . They were spaced at least 3  $\mu\text{m}$  apart to avoid any mutual interaction.

## 185 3. Results

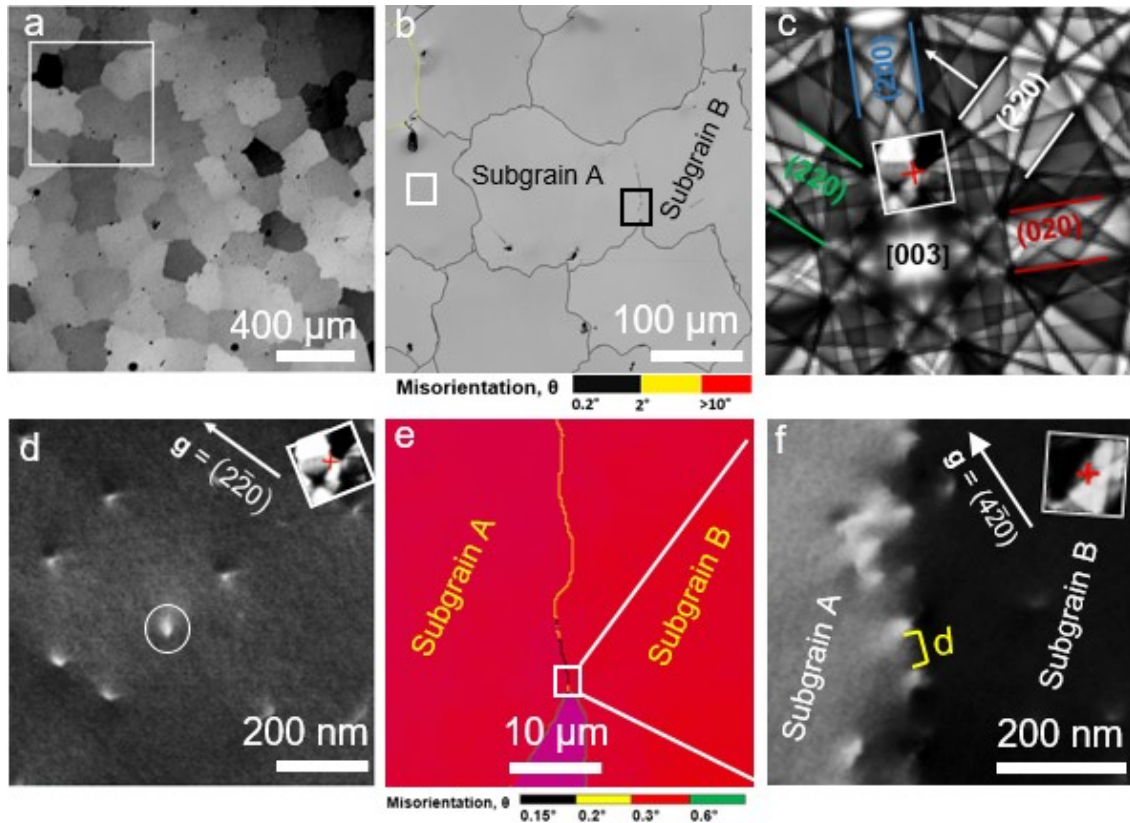
### 186 3.1. Microstructure before nanoindentation

#### 187 3.1.1. Microstructure overview of the single crystal

188 The equiatomic CrCoNi MEA crystallizes with a single-phase FCC structure [9].  
189 A representative BSE micrograph (Fig. 1a) of the sample showcases various  
190 randomly oriented dendrites with variable sizes (average: ~100  $\mu\text{m}$ ) and  
191 contrasts. Such a dendritic morphology is also referred to as crystal mosaicity in

192 the literature and was, for example, observed in Ni-base superalloy single crystals  
193 grown by the same technique [50,53].

194 An HR-EBSD map was acquired for the area marked with a white frame in Fig. 1a  
195 for detailed investigation. The corresponding misorientation map in Fig. 1b  
196 reveals that the misorientation angles ( $\theta$ ) between neighboring dendrites can be  
197 as small as  $0.2^\circ$  and that most of them are lower than  $2^\circ$ . Therefore, these  
198 dendrites are separated by LAGBs since the misorientations are lower than  $10^\circ$   
199 [26]. These dendrites are also referred to as subgrains. To obtain a precise  
200 orientation of the subgrain marked with a white frame in Fig. 1b, the procedure  
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  
205 micrograph shown in Fig. 1d was acquired with the diffraction vector  $\mathbf{g} = (2\bar{2}0)$ .  
206 Figure 1d shows asymmetrical wing-shaped spots with a black and white (B-W)  
207 contrast. Each spot corresponds to a trace on the surface of a threading  
208 dislocation (TD), *i.e.*, a dislocation nearly perpendicular to the surface. Moreover,  
209 there is a highly inclined dislocation that appears with a comet-like contrast (white  
210 circle in Fig. 1d). The same area was imaged with  $\mathbf{g} = (220)$  and a similar number  
211 of dislocations were observed.



212

213 Fig. 1. (a) BSE micrograph of an [001]-oriented single-crystal of the CrCoNi alloy.  
 214 (b) Grain boundary misorientation map of the white-framed region in (a). (c) High-  
 215 resolution selected area channeling pattern (HR-SACP) from the white frame in (b)  
 216 superimposed onto a simulated Kikuchi pattern where the red cross marks the optical  
 217 axis of the SEM. (d) ECC micrograph taken inside the subgrain highlighted with a white  
 218 frame in (b) using  $\mathbf{g} = (2\bar{2}0)$ , see white arrow in (c). (e) Grain boundary misorientation  
 219 map of the black-framed region in (b) superimposed onto a subgrain orientation map.  
 220 Here, ECC analysis was performed at the region framed in white. (f) ECC micrograph of  
 221 the LAGB under  $\mathbf{g} = (4\bar{2}0)$ . 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.

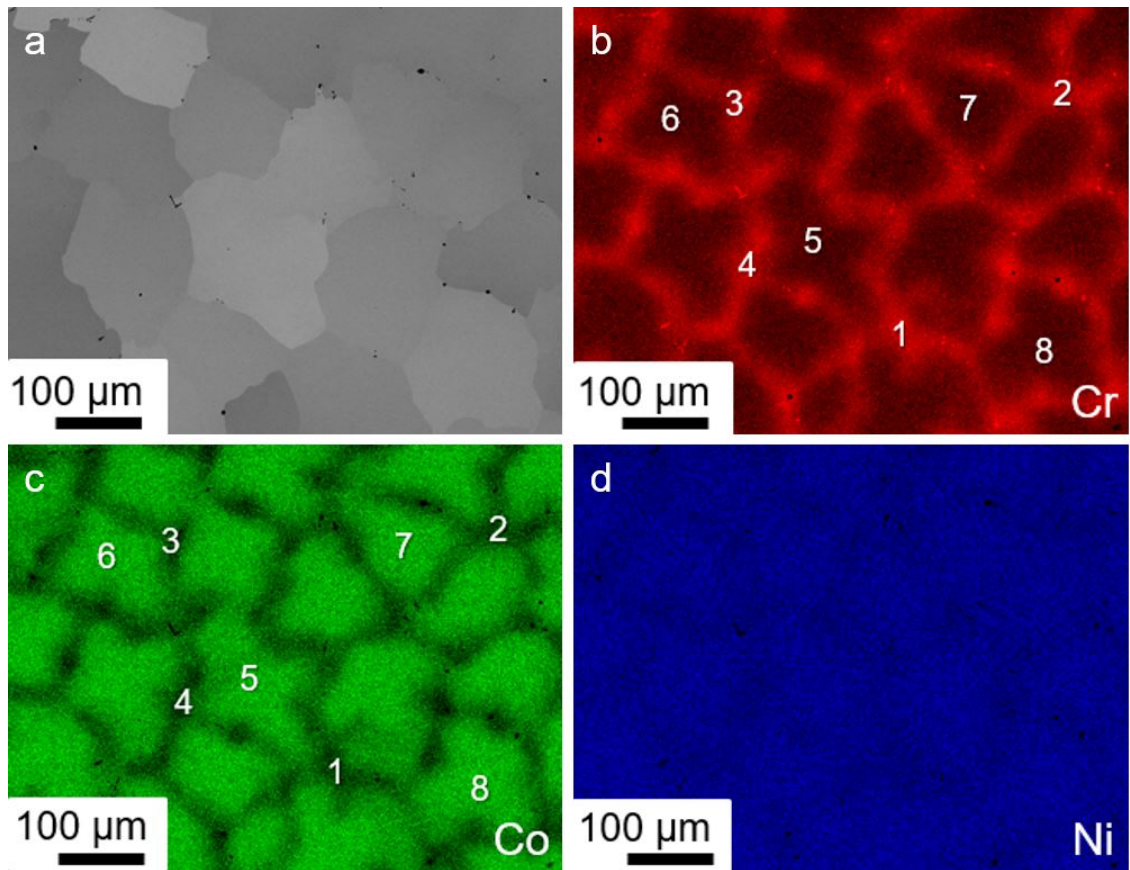
223 The TDs offer an advantage over curved or straight dislocations [54] because  
 224 they allow a better estimation of the dislocation density ( $\rho$ ) using Eq. (1):

225 
$$\rho = \frac{N}{A} \quad (1),$$

226 where  $N$  is the number of TDs and  $A$  is the surface area of the micrograph [47].  
227 The  $\rho$  value averaged over 20 micrographs (total area: 140  $\mu\text{m}^2$ ) taken inside five  
228 different subgrains is  $(3.6 \pm 0.9) \times 10^{12} \text{ m}^{-2}$ . This value is comparable to that  
229 found in as-cast single crystals [55].

230 [Figure. 2a](#) shows a BSE micrograph where several subgrains/dendrites can be  
231 observed. The EDX maps shown in [Figs. 2b-d](#) display the distribution of chemical  
232 elements within the dendritic microstructure and the results of EDX point  
233 analyses, performed at locations highlighted by the numbers 1-4 in interdendritic  
234 and 5-8 in dendritic regions, are summarised in [Table 1](#), respectively. The  
235 dendrites are enriched in Co and depleted in Cr with an average composition of  
236  $\text{Cr}_{30}\text{Co}_{36}\text{Ni}_{34}$  and have a size of  $\sim 100 \mu\text{m}$ . In contrast, interdendritic regions are  
237  $\sim 25 \mu\text{m}$  wide with an average composition of  $\text{Cr}_{35}\text{Co}_{32}\text{Ni}_{33}$ .

238



239

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

242 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	Co	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

244

245 Since all our nanoindentations were performed at distances that are at most 12  $\mu\text{m}$   
246 away from the LAGB (see Fig. 3a), the mechanical responses are therefore  
247 representative of the composition present in the interdendritic regions  
248 ( $\text{Cr}_{35}\text{Co}_{32}\text{Ni}_{33}$ ).

### 249 3.1.2. Structure of the LAGB of interest

250 The region marked with a black frame in Fig. 1b is magnified in the crystallite  
251 orientation map shown in Fig. 1e where the red color indicates that the crystallites  
252 have near  $\{001\}$  orientations. Figure 1e is centered on a LAGB of interest that  
253 separates subgrains A and B. The range of  $\theta$ -values along the LAGB is  $0.15^\circ$ - $0.3^\circ$   
254 as deduced from the misorientation map. Assuming that this LAGB consists of an  
255 array of equally spaced dislocations, invoking the model of Read and Shockley  
256 [55], the expected spacing between dislocations ( $d$ ) can be estimated using:

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

258 where  $\mathbf{b} = \frac{1}{2} a \langle 110 \rangle$ . Using  $\theta = 0.15$ - $0.3^\circ$  in combination with Eq. 2, we find  
259  $d$  values between 100 and 50 nm. The ECC micrograph (Fig. 1f) acquired with  
260  $\mathbf{g} = (4\bar{2}0)$  in the white-framed region of Fig. 1e shows that the LAGB consists of  
261 an array of TDs with a mean spacing  $d = 60 \pm 5$  nm (see the yellow bracket in  
262 Fig. 1f). From this value, an average misorientation of  $0.24 \pm 0.02^\circ$  can be  
263 deduced from Eq. 2. This latter value is consistent with the misorientation angle  
264 determined by EBSD and validates our approach using A-ECCI.

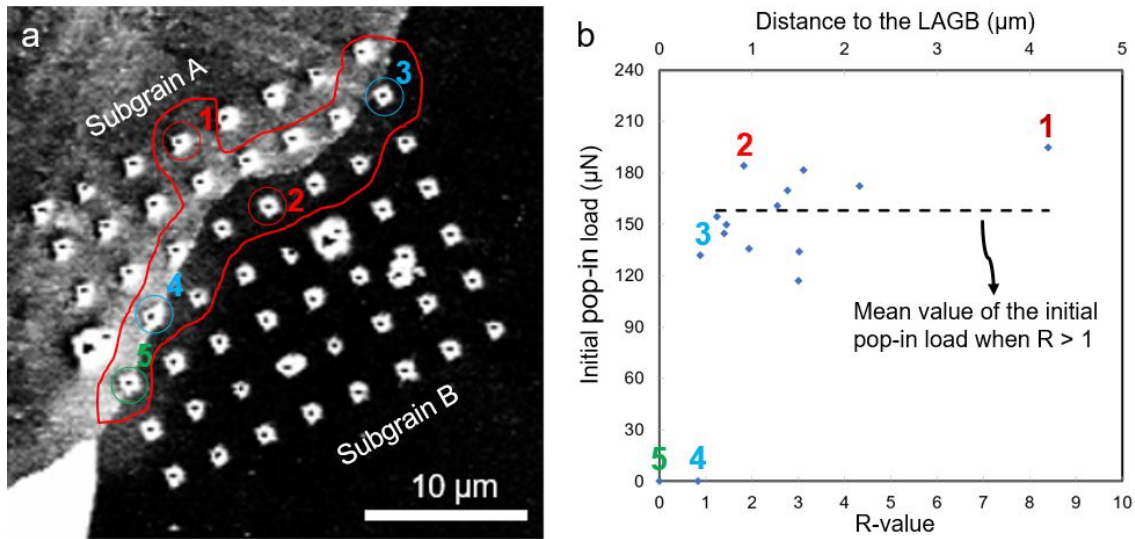
265 Due to the surface relaxation effect, the Burgers vectors of the TDs within grains  
266 in Fig. 1d and along the LAGB in Fig. 1f cannot be determined using the invisibility  
267 criteria  $\mathbf{g} \cdot \mathbf{b} = 0$  and  $\mathbf{g} \cdot \mathbf{b} \times \mathbf{u} = 0$ , where  $\mathbf{u}$  is a unit vector along the dislocation



268 line [44-45,56-57] because these dislocations exhibit a residual contrast even if  
269 the invisibility criteria are fulfilled. Therefore, it is not straightforward to determine  
270 whether a particular dislocation has an edge, mixed, or screw character.  
271 However, Tunstall and Hirsch [56] showed by theoretical calculations and TEM  
272 imaging in Pt and Cu that the B-W contrast of TDs is stronger and laterally larger  
273 for screw TDs than edge TDs. As the TDs along the LAGB (Fig. 1f) show varying  
274 contrasts, this implies that the boundary has a mixed tilt and twist character.

### 275 3.2. Mechanical response and microstructure after 276 nanoindentation

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



292

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

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

### 304 3.2.1. Nanoindentation inside subgrains

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

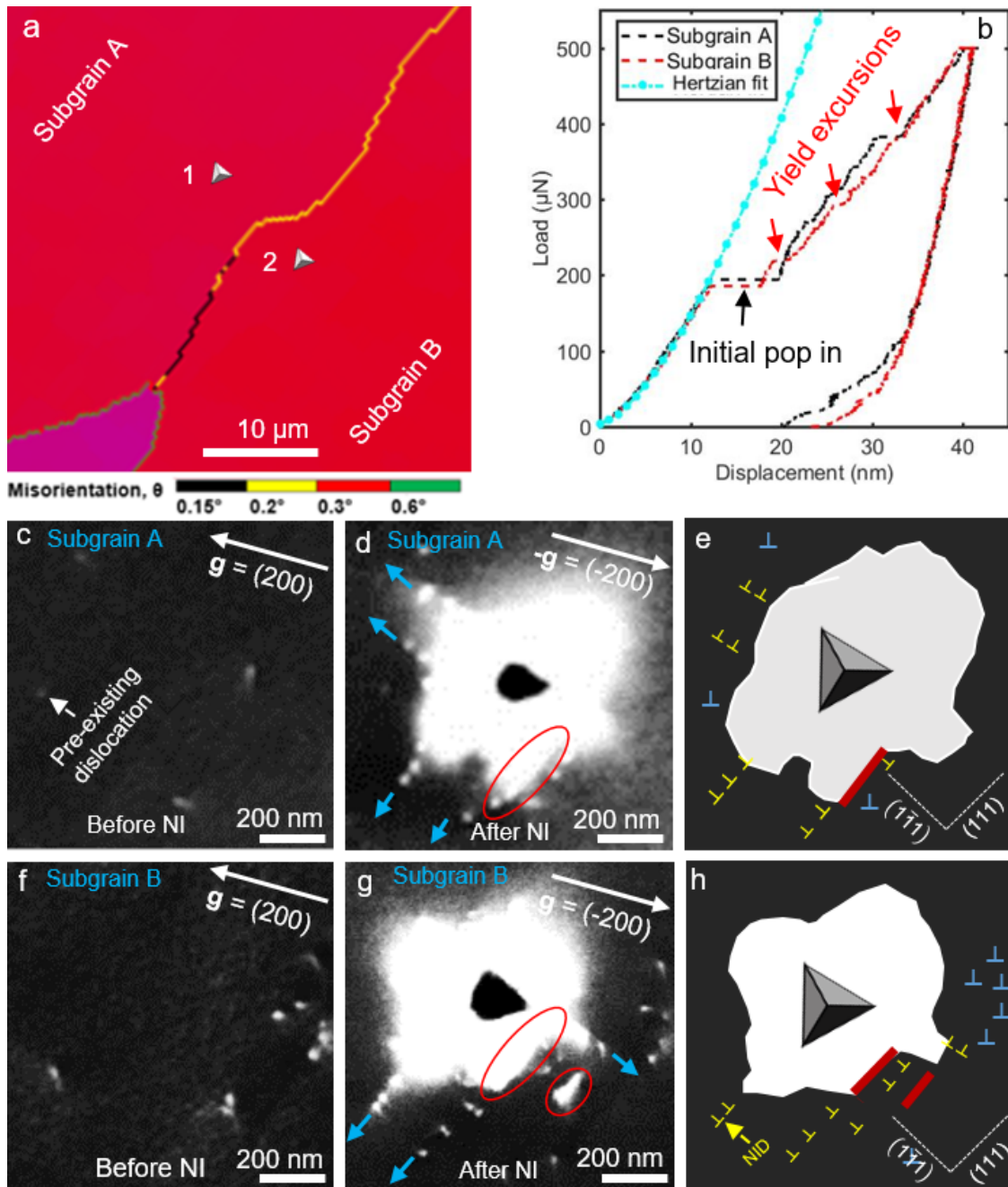


309 ( $\rho = 2.02 \times 10^{12} \text{ m}^{-2}$ ). Their load-displacement ( $P-h$ ) curves are shown for  
310 subgrains A and B as black and red curves in Fig. 4b, respectively. They exhibit  
311 an initially purely elastic loading that can be described by the Hertzian contact  
312 theory, see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

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

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

352 A similar microstructural evolution was observed when nanoindentation was  
353 performed in subgrain B (Figs. 4f-h). However, the average length of the pile-ups  
354 on the (111)/(11 $\bar{1}$ ) planes was longer ( $0.73 \mu\text{m}$ ) than that in subgrain A. In  
355 contrast, the mean length of the pile-ups on the ( $\bar{1}\bar{1}1$ )/( $\bar{1}\bar{1}\bar{1}$ ) planes was shorter  
356 ( $0.55 \mu\text{m}$ ) and these pile-ups are located on the opposite side of the indent-



358

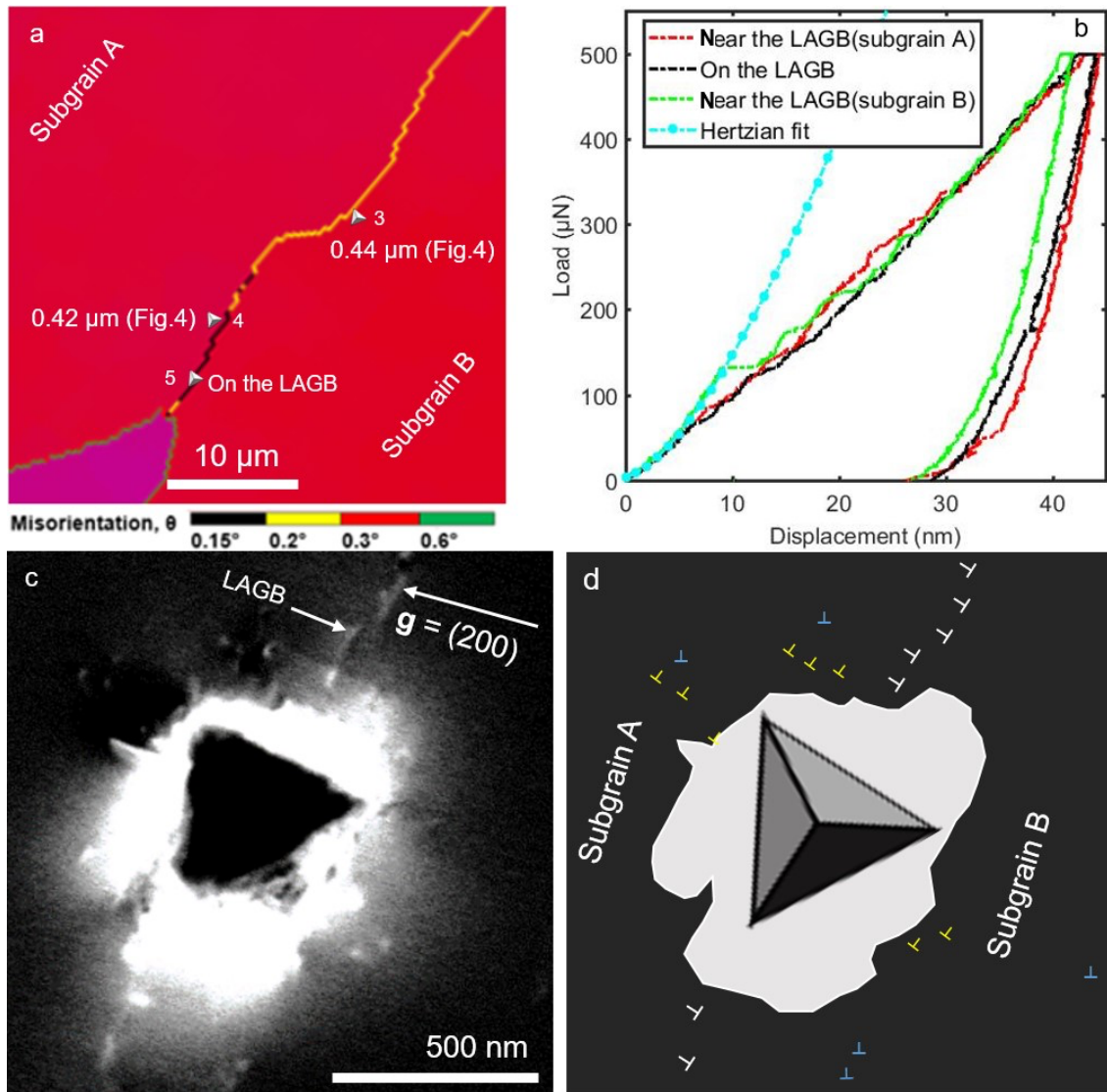
359 Fig. 4. (a) Positions of the indents in the subgrains A and B superimposed onto the EBSD  
 360 map (indents #1 and #2 in Fig. 3a). (b) Load-displacement curves in subgrains A and B.  
 361 The initial elastic loading stage follows the Hertz contact theory. (c-h) Microstructural  
 362 evolution as a result of nanoindentation in regions with a low initial local dislocation  
 363 density ( $\rho = 2.02 \times 10^{12} \text{ m}^{-2}$ ). (c-d) are ECC micrographs before and after

364 nanoindentation in the subgrain A, respectively. (e) is a schematic of (d). (f-g) are ECC  
365 micrographs before and after deformation within subgrain B. (h) is a schematic of (g).  
366 The white dashed lines in (e and h) show the traces of the  $(111)/(11\bar{1})$  and  $(\bar{1}\bar{1}1)/(\bar{1}\bar{1}\bar{1})$   
367 planes. The red ellipses in (d) and (g) and red thick lines in (h) and (e) mark the traces  
368 of SFs. NI and NID stand for nanoindentation and nanoindentation induced dislocation,  
369 respectively. Blue arrows in (d) and (g) show pile-ups directions. Note that the symbol  $\perp$   
370 is used to mark the positions of dislocations and does not refer to their character.

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

### 375 3.2.2. Nanoindentation on and near the LAGB

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



388

389 Fig. 5. (a) Positions of the indents superimposed on the subgrain orientation map.  
 390 (b) Load-displacement curves of nanoindentation tests performed at the LAGB (#5),  
 391 0.42  $\mu\text{m}$  ( $R = 0.84$ ) from the LAGB in subgrain A (#4), and 0.44  $\mu\text{m}$  ( $R = 0.88$ ) in subgrain  
 392 B (#3). The initial elastic loading follows the Hertz contact theory (light blue curve).  
 393 (c) ECC micrograph of indent #5 performed at the LAGB and (d) corresponding  
 394 schematic. Note that the symbol  $\perp$  is used to mark the positions of dislocations and does  
 395 not refer to their character.

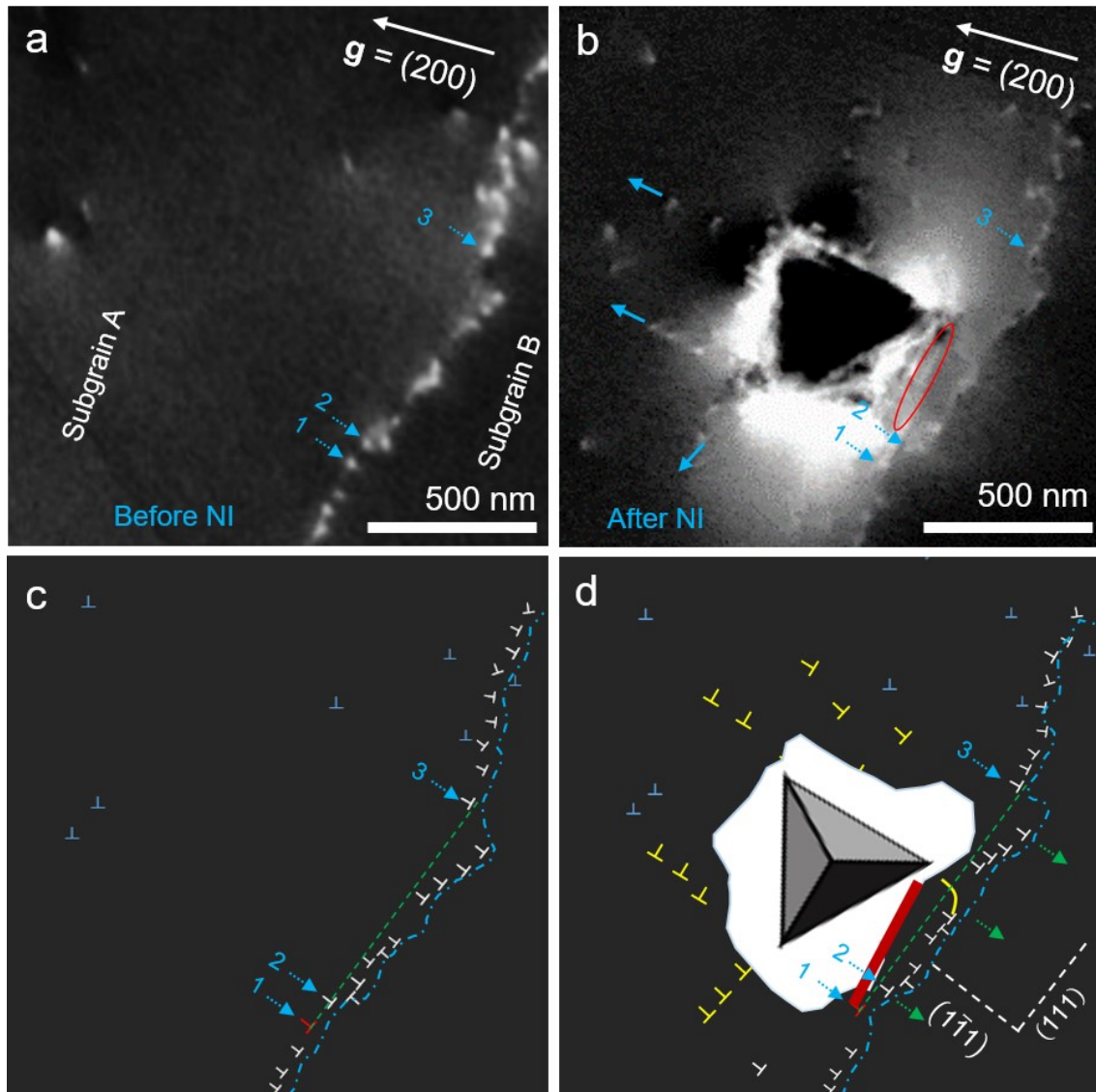
396 To investigate the interactions of dislocations with the LAGB, nanoindentation  
 397 tests were performed at  $\sim 0.42 \mu\text{m}$  ( $R = 0.84$ ) from the LAGB in the subgrain A

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

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



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

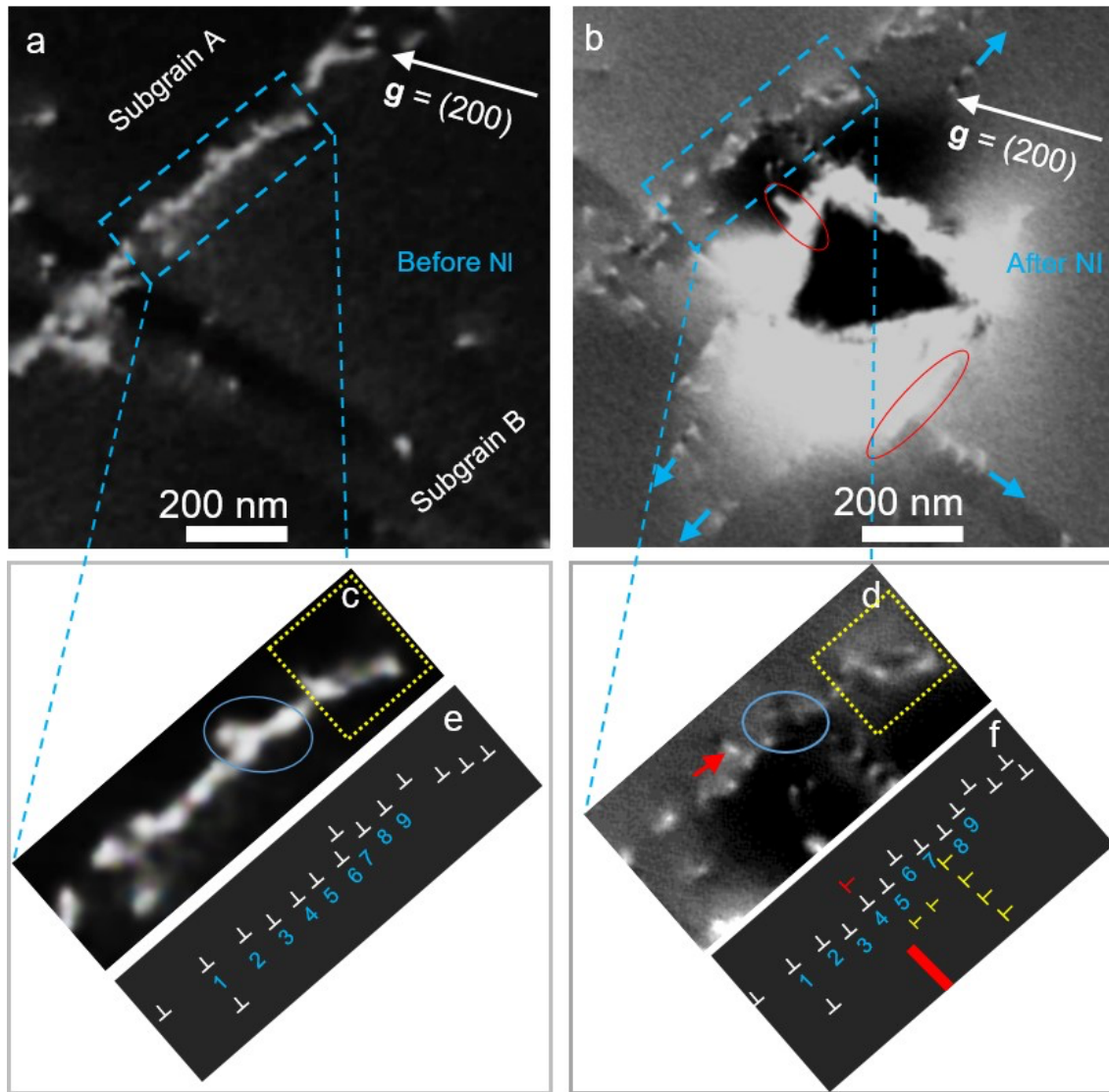


444

445 Fig. 6. (a-b) ECC micrographs before and after nanoindentation showing the evolution  
 446 of the dislocation configurations in the subgrain A at  $0.42 \mu\text{m}$  ( $R = 0.82$ ) from the LAGB  
 447 (indent #4 in Fig. 3a). (c-d) schematics of (a-b). The red ellipsis in (b) and red line in (d)  
 448 show an SF. The blue arrows in (b) show directions along which the NID pile-ups extend.  
 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  
 451 dislocations 1, 2, and 3 before and after nanoindentation while the green arrows show  
 452 the direction of the slight motion of LAGB dislocations. Note that the symbol  $\perp$  is used to  
 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  
455 nanoindentation for the indent performed in the subgrain B at  $R = 0.88$ . It displays  
456 pile-ups of NIDs (blue arrows in [Fig. 7b](#)) and two SFs (red ellipses in [Fig. 7b](#)) on  
457  $\{111\}$  planes. The lengths of the pile-ups of NIDs are  $0.63\ \mu\text{m}$  and  $0.57\ \mu\text{m}$  on  
458 the traces of  $(111)/(11\bar{1})$  and  $(1\bar{1}1)/(1\bar{1}\bar{1})$  planes, respectively. Those on  
459  $(1\bar{1}\bar{1})/(1\bar{1}\bar{1})$  were expected to interact with the LAGB and a change of the  
460 dislocation structure of the LAGB was indeed observed as a result of the  
461 nanoindentation test. To better observe these evolutions, high-resolution ECC  
462 micrographs were recorded before and after nanoindentation at the location  
463 marked with a blue frame in [Figs. 7a-b](#). These micrographs are displayed in  
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  
466 numbered from one to nine. As mentioned before, please note that the resolution  
467 of [Figs. 7b,d](#) is higher than that used for [Figs. 7a,c](#). After nanoindentation, NIDs,  
468 shown as yellow dislocations in [Fig. 7f](#), can be observed at  $\sim 40\ \text{nm}$  from the  
469 LAGB. These NIDs caused the rearrangement and the collective motion of the  
470 pre-existing LAGB dislocations. The rearrangement is mainly visible between the  
471 LAGB dislocations 5 and 8. Here, the dislocations form a “>” before  
472 nanoindentation that expanded after nanoindentation (compare ellipses in  
473 [Figs. 7c and 7d](#)).



474

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

481 The local collective motion of the LAGB dislocations between 1 and 9 away from  
 482 the residual indent (toward the subgrain A) is shown in the animated  
 483 superimposition of micrographs before and after nanoindentation, respectively, in

484 [Fig. S2 \(supplementary data\)](#). Note that the two dislocations located in subgrain  
485 A on the left of [Figs. 7a,b](#) were used as markers for this GIF file.

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

## 500 4. Discussion

501

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

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

527 Interestingly the indents #5 and #4 performed on the LAGB and at  $R = 0.84$  in  
528 subgrain A, respectively, do not exhibit pop-ins (see Fig. 3) and deviate at low  
529 loads from the Hertz law, even though they show different microstructure  
530 evolutions during nanoindentation. For indent #4 at  $R = 0.84$ , the absence of  
531 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  
533 sample surface and deeper than the extinction depth of ECCI (~100nm), the  
534 stress field below the indenter may have interacted with the inclined dislocations  
535 of the LAGB, and plastic deformation was accommodated by the motion and  
536 multiplication of these pre-existing LAGB dislocations.

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

## 547 5. Conclusions

548

549 The following conclusions can be drawn from our findings. The LAGB of interest  
550 has an average misorientation of  $0.24^\circ$  and consists of dislocations aligned along  
551 the [001] direction with a mean spacing of ~60 nm. Besides, the elemental  
552 composition in the vicinity of this LAGB is  $\text{Cr}_{35}\text{Co}_{32}\text{Ni}_{33}$  (in at.%) in a region that  
553 is ~25  $\mu\text{m}$  wide while the subgrains (size: ~100  $\mu\text{m}$ ) have an average composition  
554 of  $\text{Cr}_{30}\text{Co}_{36}\text{Ni}_{34}$ . When nanoindentation tests are performed within subgrains with  
555 a maximum load of 500  $\mu\text{N}$ , pile-ups of NIDs form on {111} planes around the

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

### 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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