

1 Reversible phase transformation in polycrystalline TRIP  
2 steels induced by cyclic indentation performed at the  
3 nanometric length scale

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15 **Abstract:** Metastable austenitic stainless steels are an interesting group of  
16 materials, which exhibit the TRansformation Induced Plasticity effect. In this  
17 regard, phase transformation from austenite to martensite enhances the work  
18 hardening of the metastable austenitic stainless steels affecting the deformation  
19 dynamics and mechanical properties including fatigue properties. Within this  
20 context, the reversible load-induced phase transformation from  $\gamma$  to  $\varepsilon$ -martensite  
21 has been investigated at the local scale under cyclic indentation. This reversible  
22 phase transformation was manifested itself by a combination of hysteresis  
23 loops, elbow formation and reversible pop-ins in the loading curve. The initial  
24 cyclic achieved through the nanoindentation technique allowed to identify three  
25 different deformation regime for the  $\langle 111 \rangle$  austenitic grains. At the first step, the  
26 dislocation activation producing a softening effect took place, subsequently the  
27 phase transformation induced a hardening effect, finally, a plateau was reached  
28 where no more plastic deformation was observed.

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30 **Keywords:** metastable austenitic stainless steels; nanoindentation; reversible  
31 phase transformation; cyclic indentation; plastic deformation mechanisms;  
32 shape memory effect.

## 33 1. Introduction

34 Metastable austenitic stainless steels are materials widely employed in the  
35 automotive industry due to their distinguished mechanical properties [1]. In this  
36 sense, the mechanical behavior is governed by a conjunction of multiple  
37 deformation mechanisms, whereby the most decisive might be the  
38 Transformation Induced Plasticity (TRIP), which is widely used to denote the  
39 austenite-to-martensite phase transformation. Thereby, this transformation can  
40 be direct (from  $\gamma$  to  $\alpha'$ ) or indirect via the intermediate hexagonal phase (from  $\gamma$   
41 to  $\varepsilon$ -martensite) [2,3,4,5]. The phase transformation effect on metastable  
42 austenitic stainless steels depends highly on the Stacking Fault Energy (SFE)  
43 [6,7]. The compositional dependence of the alloy is an important factor affecting  
44 SFE and it can be determined by using the empirical equation proposed by  
45 Schramm and Reed [8]:

$$46 \text{ SFE} = -53 + 6.2 (\text{Ni}) + 0.7 (\text{Cr}) + 3.2 (\text{Mn}) + 9.3 (\text{Mo}) \quad (1)$$

47 where SFE is in  $\text{mJ}\cdot\text{m}^{-2}$  and the elements present in equation (1) are in mass  
48 %. Thin  $\varepsilon$ -martensite plates can be formed easily from planar austenitic defects,  
49 since its structure corresponds to an overlapping of intrinsic stacking fault (SF)  
50 on every second  $\{111\}$  plane as reported in Refs. [9,10]. The reversible phase  
51 transformation ( $\gamma \Leftrightarrow \varepsilon$ ) is well exploited in shape memory alloys such as TiNi  
52 [11] or Fe-Mn-Si-Cr [12]. On the other hand, in Fe-Ni-Cr based metastable  
53 austenitic stainless steels,  $\varepsilon$ -martensite is considered as unstable transition  
54 phase, which further transforms to  $\alpha'$ -martensite [13,14]. In this regard,  $\varepsilon$ -  
55 martensite may be considered as a dislocation pile-up of intrinsic stacking faults  
56 (SFs) on every second (111) austenite plane, the  $\gamma \Rightarrow \varepsilon$  transformation is a  
57 rather simple process. Recently, Roa *et al.* [15] observed the  $\gamma \Rightarrow \varepsilon$  martensitic  
58 phase transformation in highly sheared austenitic zones, mainly located at the  
59 grain boundary.

60 In this context, the aim of the present paper is to locally study and correlate the  
61 shape and the different bursts of the cyclic loading-unloading curves by means  
62 of the nanoindentation technique. The reversible phase transformation will be  
63 investigated in order to allow better understanding of the  $\gamma \Rightarrow \varepsilon$  martensitic  
64 transformation for metastable austenitic stainless steels.

## 65 **2. Experimental procedure**

66 The material investigated in this work was a commercial annealed AISI 301LN  
67 stainless steel at 1100°C for 1h, equivalent to EN 1.4318, supplied by  
68 Outokumpu (Finland) as 2 mm thick sheets. The chemical composition is shown  
69 in **Table 1**. Prior the microstructural and micromechanical characterization, the  
70 TRIP steel specimens were polished with silicon carbide and then diamond  
71 suspension of 30, 6, 3 and 1  $\mu\text{m}$ . Finally, a neutral suspension of 20 nm alumina  
72 particles was used in order to remove possible work hardening introduced  
73 during surface preparation. The average grain size for the austenitic phase was  
74 directly determined by the linear intercept method yielding a monomodal  
75 austenitic equiaxial grains distribution, whose sizes are  $12.0 \pm 2.4 \mu\text{m}$ .

76 The ultra-nanohardness tester (UNHT) from CSM instruments using a  
77 Berkovich tip indenter was employed to perform cyclic nanoindentation tests  
78 working under loading mode at a maximum applied load of 6 mN. The indenter  
79 shape has been carefully calibrated for true indentation depth as small as 20  
80 nm by indenting fused silica samples of known Young's modulus (72 GPa).  
81 Furthermore, the loading and unloading rates were held constant and equals to  
82  $15 \text{ mN}\cdot\text{min}^{-1}$  for all the tests.

## 83 **3. Results and discussion**

84 Loading-unloading (P-h) curves for 50 cycles are depicted in **Figure 1a**, which  
85 were conducted between 10% of the peak working under load control mode and  
86 6 mN of maximum applied load. In this representation, each cycle has been  
87 shifted 10 nm in order to observe the real cycle shape. A pop-in or a sudden  
88 displacement-burst (see black arrow) at constant load of around 0.2 mN is  
89 evident in the first loading cycle. As it is well established in the Hertzian contact  
90 theory [16], prior to the first pop-in, the deformation behavior is purely elastic.  
91 This phenomenon is well understood for a wide variety of materials (e.g. metals,  
92 ceramics, coatings, *etc.*) and it may be related mainly to the first stage where  
93 the sample initially undergoes a plastic deformation as found in Refs.  
94 [17,18,19,20,21,22]. As it is evident in this representation, no stress relaxation  
95 or softening mechanisms takes place due to all the P-h cycles reach the same  
96 maximum applied load, around 6 mN. A magnification of several loading-

97 unloading cycles (N) is represented in **Figure 1b**, where the maximum  
98 deformation takes place for the initial P-h cycles; when the indentation cycle  
99 increases, the aperture of the P-h cycles decreases. At the macroscopic length  
100 scale, when the TRIP steels are investigated under conventional dynamic tests,  
101 a similar phenomenon is appreciated [23,24,25,26,27,28,29], which is mainly  
102 attributed to the ratcheting strain accumulation effect. Within this context, the  
103 open loops for low cyclic cycles may be described as a consequence of  
104 increasing the dislocation activity during the indentation process. The width  
105 between the loading and unloading curve decreases until reaching a saturation  
106 level after 30 indentation cycles from which it remains invariant. This trend  
107 highlights the difficulty to activate the dislocation motion for the same applied  
108 stress. For this reason, the P-h curve presented in **Figure 1a** does not present  
109 a softening or hardening effect.

110 The load-displacement curves exhibit multiple small serrations as clearly  
111 evident in **Figure 2b** referred as pop-ins and pop-outs to occur during loading  
112 and unloading, respectively and are clearly observed for the <111> austenitic  
113 grains as shown in **Figure 2**.

114 **Figure 2a**, illustrates a cyclic load-penetration depths curve conducted under  
115 loading control mode between 10% of the peak loading value and the maximum  
116 applied load, 6 mN. As it is evident, a large plastic deformation takes place  
117 during the initial cycle followed by a fully elastic behavior for cycles 2 to 7 where  
118 the maximum stress applied remains constant and no softening or hardening  
119 mechanisms takes place. Afterwards, the aperture at the center of the P-h  
120 curves started to increase for the subsequent cycles (from cycle 7 to 10), where  
121 a softening effect takes place followed by a hardening behavior above cycle 11,  
122 labelled as S and H, respectively in **Figure 2a**.

123 In the bottom part of the P-h curve for cycles 6 and 8 the unloading curve  
124 abnormally bends and the loading and unloading curve intersect at that point  
125 (see dash circles and labelled as (1) and (2)). This gradual change in the slope  
126 is well known as elbow, which indicates an activation of a reversible plasticity or  
127 pseudoplasticity. This phenomenon, which is well studied and understood for Si  
128 and other semiconductor materials at the nanometric length scale, had never  
129 been observed before for TRIP steels. This behavior can be related to three

130 different factors: (i) reversible amorphization under load [30,31,32], (ii)  
131 annealing treatments [33], and (iii) reversible phase transformation  
132 [34,35,36,37]. Pan *et al.* [11] by means of cyclic nanoindentation tests on NiTi  
133 shape memory alloys observed the same phenomenon described above, which  
134 may be related to stress-induced martensitic transformation during the  
135 unloading process. This reversible transformation was accompanied by an  
136 anelastic re-loading curves with elbow formations, such as those observed in  
137 **Figure 2a**. As it is evident in this figure, a change of material response under  
138 cyclic indentation takes place during the 9<sup>th</sup> cycle, while during the previous  
139 cycles the behavior is fully reversible. Furthermore, the deformation induced  
140 during the first indentation cycle contributes to increases the critical stress for  
141 further slip and phase transformation, which becomes more favorable during the  
142 subsequent P-h cycles. This observation is similar to that observed for the cycle  
143 9 and 11, just after the elbow observation. A magnification of several open P-h  
144 cycles, showing an anelastic behavior, which may be related to the reversible  
145 phase transformation,  $\gamma \leftrightarrow \varepsilon$ , during the loading process is depicted in **Figure**  
146 **2b**. The shape memory effect might be too weak to be observed for TRIP steels  
147 by conventional cyclic techniques. However, cyclic nanoindentation technique  
148 induces dislocation motion as well as shear bands for TRIP steels, favoring the  
149 phase transformation over dislocation slip [38]. Furthermore, it facilitates  
150 reversible  $\gamma \leftrightarrow \varepsilon$  transformation, rather than  $\gamma \Rightarrow \alpha'$  or even  $\varepsilon \Rightarrow \alpha'$   
151 transformation. After the second elbow (labelled as (2)), and anelastic behavior  
152 takes places. After that, the cycle is open and a high density of pop-ins and  
153 pop-outs are clearly visible, as depicted in **Figure 2b** and also in inset of **Figure**  
154 **2c**. Furthermore, above the 11<sup>th</sup> cycle, a hardening mechanisms is activated  
155 due to the plastic strain accumulated during the previous cyclic P-h curves [39]  
156 as well as the direct  $\alpha'$ -martensite transformation from  $\gamma$  without  $\varepsilon$ -martensite as  
157 reported in Ref. [40]. After the 12<sup>th</sup> cycle, the P-h curve did not exhibit any major  
158 shape change. On the other hand, the maximum applied load in each  
159 subsequent cycle slightly increases and the pop-ins and pop-outs become more  
160 pronounced.

161 In **Figure 2c**, the experimental as well as the modified curves (where the pop-  
162 ins and pop-outs are removed and the continuous parts of the P-h curve are

163 stitched together) of the last P-h cycle is represented. Both representations  
164 present a similar stiffness ( $S = dP/dh \approx 0.14 \text{ mN}\cdot\text{nm}^{-1}$ ) without elbow  
165 phenomena. The inset exhibits a magnification of the P-h curve, where the pop-  
166 ins and pop-outs are clearly discerned. Furthermore, the sum of all pop-ins is  
167 around the same that the sum of the different pop-outs,  $\sum \Delta h_{\text{pop-in}} \approx \sum \Delta h_{\text{pop-out}} \approx$   
168 25 nm. This finding indicates that the dislocations generated during the loading  
169 process are annihilated during the unloading one emitted from the contact edge  
170 between the Berkovich indenter and the  $\langle 111 \rangle$  austenitic grain. Furthermore, it  
171 is found that the load after each pop-in is relatively well described by the elastic  
172 loading equation ( $P \approx C \cdot h^{3/2}$  [16]) which means that these pop-ins are not  
173 related to the elasto-to-plastic transition. It is reasonable to assume that those  
174 pop-ins events have a different origin, mainly related to phase transformation.  
175 Furthermore, the appearance of the different pop-ins observed in the loading  
176 curve indicates the occurrence of several deformation mechanisms at a  
177 constant displacement, such as reversible dislocation gliding or by reversible  
178 phase transformation. Pop-ins arising beyond the elasto-to-plastic transition in  
179 TRIP steels have been observed by several authors under monotonic tests at  
180 the nanometric length scale and are often attributed to phase  $\gamma \Rightarrow \alpha'$  phase  
181 transformation [41,42]. Thus, as it is depicted in **Figure 2c**, the different pop-ins  
182 are reversible. The similar trend was observed by Ahn *et al.* [43], where multiple  
183 pop-ins were observed in the loading curve and these features were attributed  
184 to the  $\gamma \Rightarrow \varepsilon$  transformation observed by transmission electron microscopy  
185 (TEM). It is worth to mention that in austenitic stainless steels the mechanically  
186 induced formation of  $\varepsilon$ -martensite is reported to occur when the SFE is below  
187  $18 \text{ mJ}\cdot\text{m}^{-2}$  [44], which is the case of the material studied in this work (SFE= $7.87$   
188  $\text{mJ}\cdot\text{m}^{-2}$ ).

189 Within this context, the  $\gamma \Rightarrow \varepsilon$  phase transformation under loading is the most  
190 widely reported reason for occurrence of pseudoelasticity in iron-based shape  
191 memory alloys [45,46,47,48], where the pseudoelasticity phenomenon is mainly  
192 observed during the cyclic loading process as reported in Refs. [12,49]. This  
193 phenomenon is mostly attributed to the reversible transformation ( $\gamma \Leftrightarrow \varepsilon$ ) under  
194 load, which can be triggered strain-induced plasticity at room temperature,  
195 particularly in alloys with low stacking fault energy, SFE [49,50,51]. On the other

196 hand, the deformation-induced  $\varepsilon$ -martensite formation in alloys with a low Ni  
197 content (see **Table 1**) has been reported to be rather unstable [52].

198 The transformation between  $\gamma$  and  $\varepsilon$  is expected to easy occur, while the  $\varepsilon \Rightarrow \alpha'$   
199 martensitic transformation requires a higher activation energy. Thus, the  
200 different pop-ins observed in the loading curve, **Figure 2c**, may be related to  
201 the phase transformation from  $\gamma$  to  $\varepsilon$ , while the  $\varepsilon \Rightarrow \alpha'$  martensitic  
202 transformation is practically impossible to occur due to the stress generated  
203 during the indentation process, which is not high enough to induce this  
204 transformation. Furthermore, the pop-outs observed during the unloading  
205 process may be related to the reversible phase transformation (from  $\varepsilon$ -  
206 martensite to  $\gamma$ ). On the other hand, Sekeido *et al.* [53] observed multiple pop-  
207 ins in the loading curve which were attributed to the  $\varepsilon$ -martensite formation.

208 However, as can be withdrawn from **Figure 2d**, there is a correlation between  
209 the cycle number with the average pop-in width ( $\Delta h_{pop-in}$ ) and the accumulative  
210 displacement due to the different pop-ins during the loading cycle ( $\sum \Delta h_{pop-in}$ ). As  
211 it is evident in this representation, the  $\Delta h_{pop-in}$  increases steadily, almost  
212 doubling the initial value; from 0.54 nm in the cycle number 10 to 0.97 nm in the  
213 cycle number 90. On the other hand, the total displacement increases more  
214 than three times, from 8.15 to 26.1 nm; especially at the initial stage, not only  
215 the width but also the number of pop-ins increases. Around the 90<sup>th</sup> cycle a  
216 saturation is reached as confirmed also by the stabilization of the hysteresis  
217 shape observed in **Figure 2d**. This observation, may be related to the  
218 accumulated plastic features induced along the entire cyclic process. According  
219 to Ahn and co-workers [43], the width of the different pop-ins, may be  
220 considered as the projection of the slip systems of Shockley partial dislocations,  
221 leading to the  $\varepsilon$ -martensite formation. In this regard, at least two SF are required  
222 for the formation of a  $\varepsilon$ -band, with a respective unit displacement of around 0.1  
223 nm depending on the certain slip system. Thus,  $\Delta h_{pop-in}$  reported in this study  
224 corresponds to the formation of around five to ten  $\varepsilon$ -platelets.

225 Nanoindentation technique was successfully used to identify the different  
226 deformation regimes. In this regard, the plot of the cyclic area versus cycle for  
227 the first 20 cycles is shown in **Figure 2e** where three different regions are

228 visible. This trend may be related with the plastic deformation features activated  
229 under the cyclic P-h process. A softening effect (Region 1) followed by a  
230 hardening one (Region 2) is clearly evident. In the first region, the softening  
231 behavior is related to the activation of negative dislocation sources emitted from  
232 the contact edge of the Berkovich indenter, which reduces the dislocation  
233 density in the deformed area and causes a local softening. This phenomenon  
234 may be related mainly to a direct phase transformation to  $\alpha'$ - without  $\varepsilon$ -  
235 martensite, as reported by Tian *et al.* [54]. Finally, when the phase  
236 transformation has been activated and the region deformed reaches the  
237 maximum accumulated strain under the Berkovich indenter (around 12 cycles),  
238 no more deformation features are able to be activated at the same time, for this  
239 reason, the cyclic area reaches a plateau.

240 In order to observe the dislocation motion during the holding process cyclic  
241 indentation loading tests with large maintaining time between loads were done.  
242 **Figure 3** shows the applied load (P, (mN)) (the specimen was loaded and  
243 unloaded between 0 and 6 mN in steps of 0.6 mN and a holding time between  
244 each step of 1800 s was implemented) and penetration depth (h, (nm)) as a  
245 function of time. It is clearly seen that the penetration depth increases with  
246 increasing time for each holding step and the opposite occurred during the  
247 unloading process. On the other hand, the total amount of displacement at  
248 maximum load and fully unload is quite similar, being the maximum  
249 displacement at a maximum applied load of around  $248 \pm 3$  nm, while the  
250 cumulative viscoplastic displacement is around  $64 \pm 2$  nm.

## 251 **Conclusions**

252 Cyclic nanoindentation tests in austenitic stainless steels have been performed  
253 in order to examine the correlation between the loading and unloading features  
254 (*i.e.* shape, pop-ins, pop-outs, *etc.*) and the reversible phase transformation.  
255 The main conclusions of this work can be summarized as follows:

- 256 (i) The metastable austenitic stainless steel, AISI 301 LN, can  
257 undergoes a reversible  $\gamma \Leftrightarrow \varepsilon$  martensitic phase transformation.
- 258 (ii) The phase transformation was anisotropic, being the  $\langle 111 \rangle$  austenitic  
259 grain parallel to the loading axis, the most favorable.



- 260 (iii) The reversible phase transformation has not been observed at the  
261 macroscopic length scale tests due to the fact that phase  
262 transformation has been found highly anisotropic.
- 263 (iv) Reversible phase transformation manifested in the loading-unloading  
264 curve by a combination of several features; hysteresis loops, elbow  
265 formation and reversible pop-ins in the loading curve.

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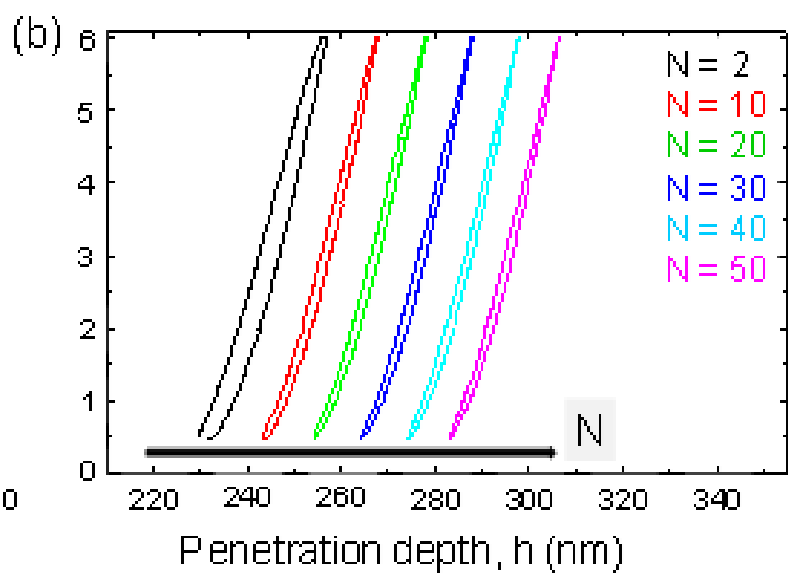
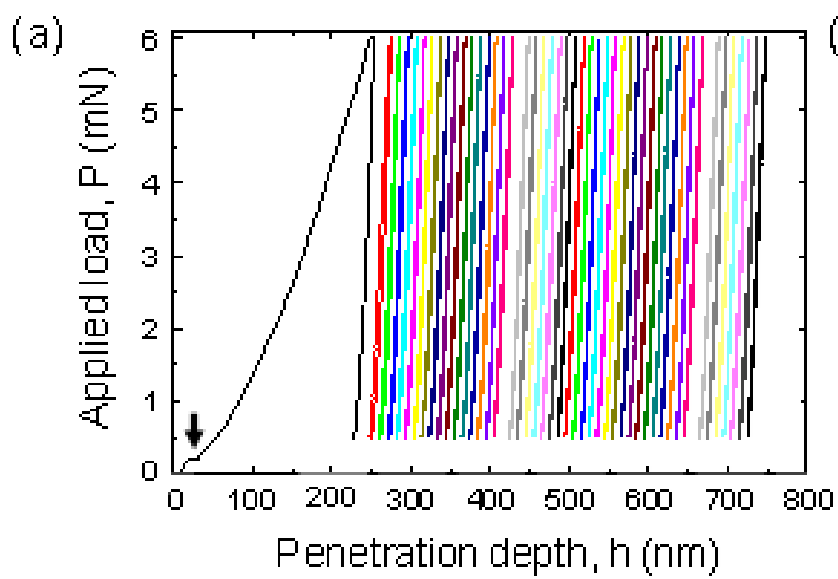
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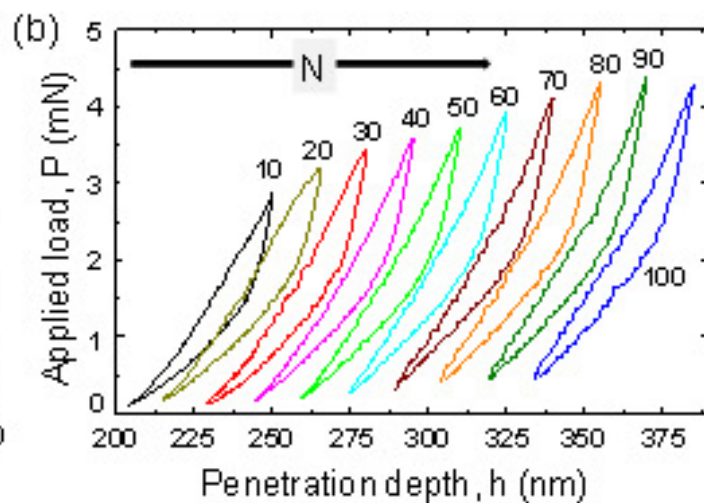
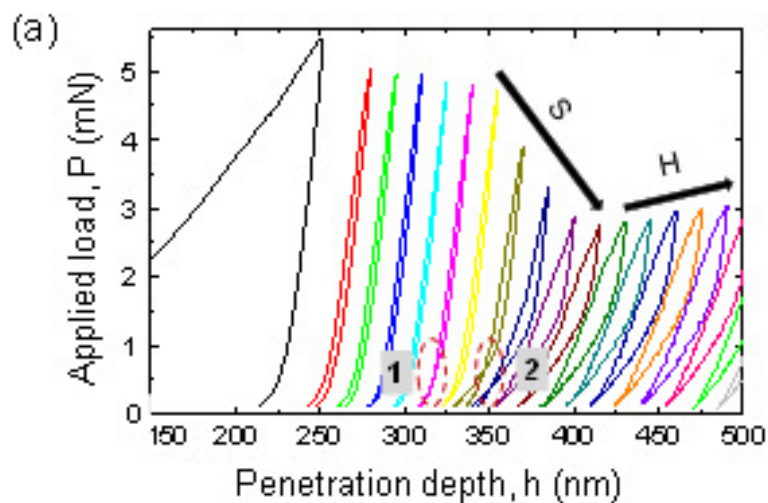
## Figure captions

**Figure 1.** (a) General view of the cyclic evolution of the P-h curves for 50 cycles performed under load control mode, which were conducted between the maximum applied load ( $\approx 6$  mN) and 10% of the peak working ( $\approx 0.6$  mN). The black arrow shows the pop-in which is attributed to the elastic-to-plastic transition and (b) detailed view of every tenth cycle. Between each cycle the penetration depth has been shifted 10 nm in order to observe better the shape of each P-h cycle.

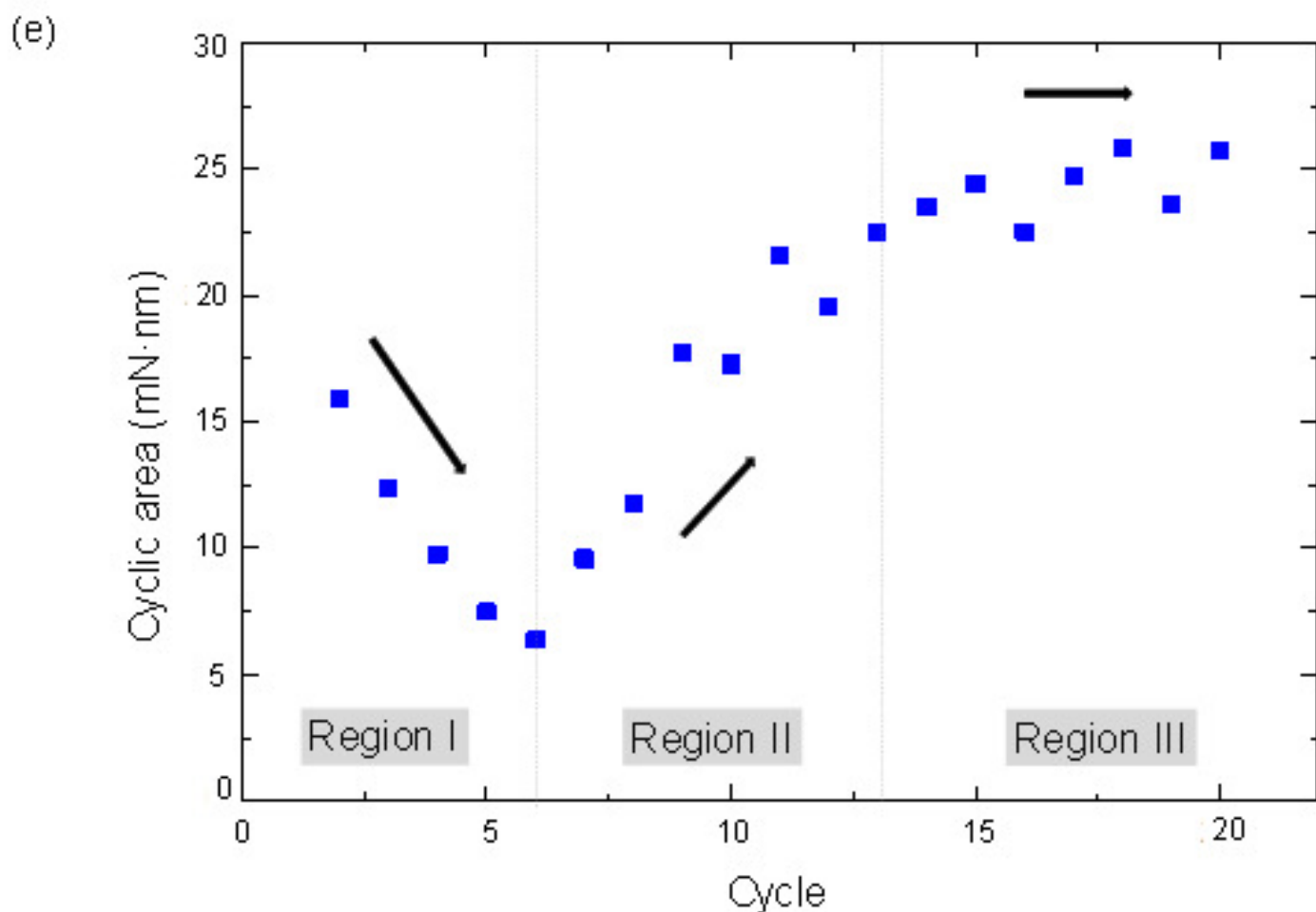
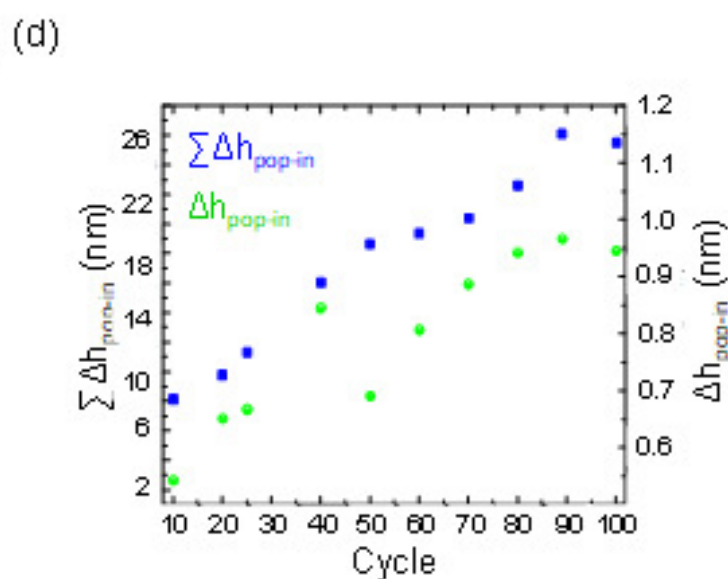
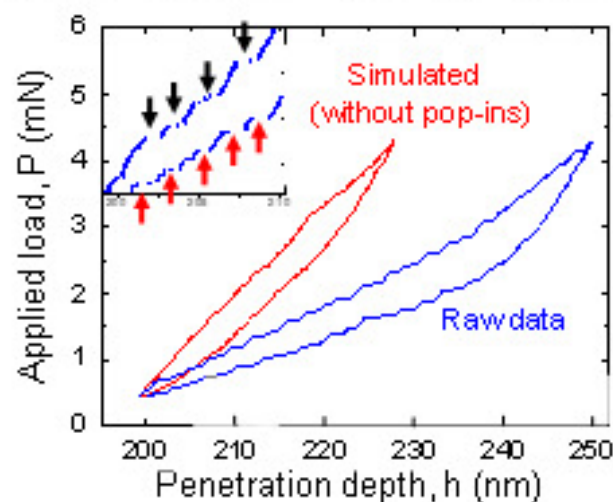
**Figure 2.** (a) General view of the cyclic evolution of the P-h curves for 100 cycles performed under load control mode at a maximum applied load of 6 mN for an annealed austenite  $\langle 111 \rangle$  grain. (b) Detailed view of every tenth cycle. Between each cycle the penetration depth has been shifted 15 nm in order to observe the shape of each P-h cycle. (c) Real and simulated (pop-in free) P-h curve for the last indentation cycle, 100. (d)  $\sum \Delta h_{\text{pop-in}}$  (blue) and  $\Delta h_{\text{pop-in}}$  (green) as a function of the cycle number and (e) Cyclic P-h area as a function of the cycle.

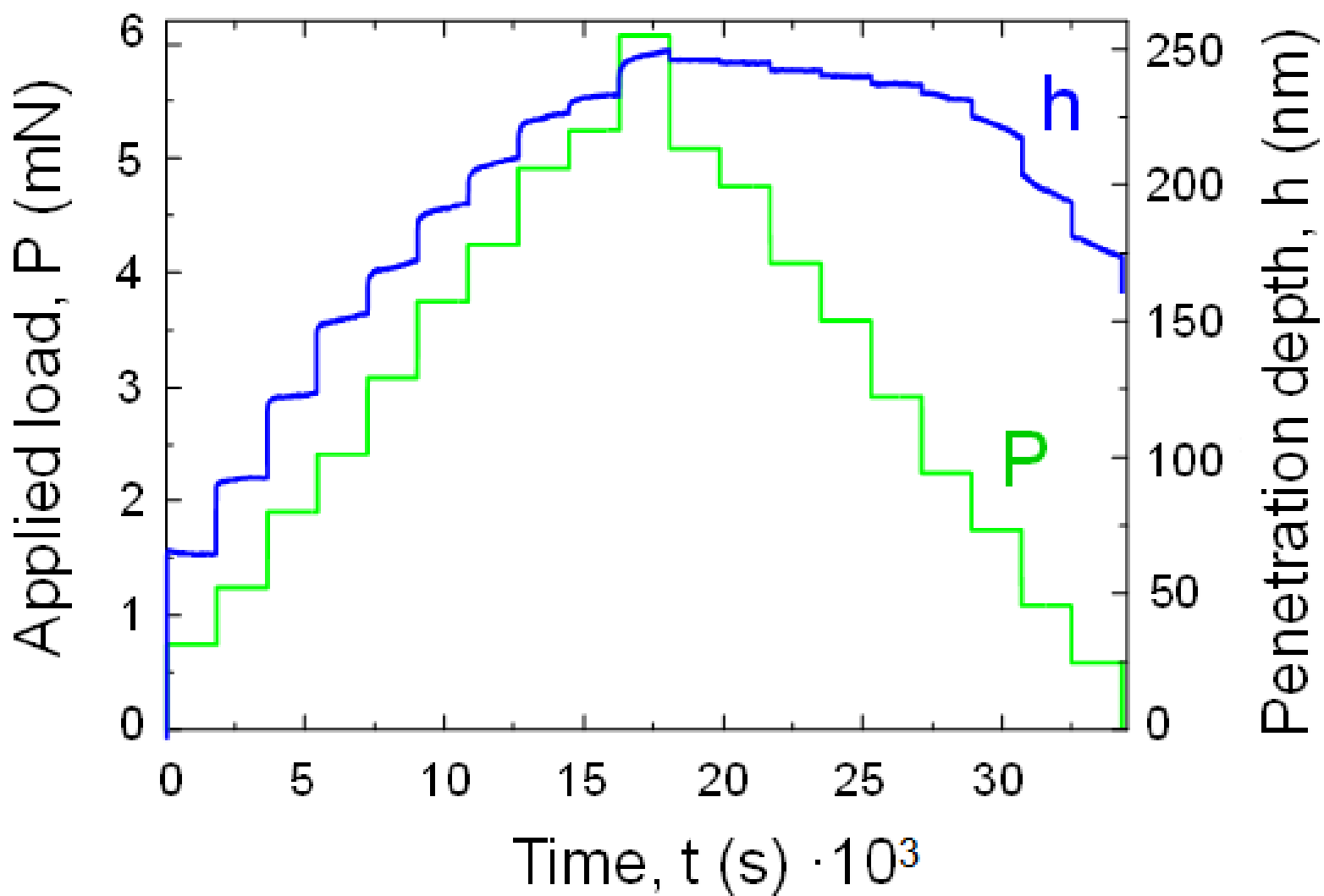
**Figure 3.** Multi-step nanoindentation testing with a holding period in each step holded constant and equals to 1800 s. Applied (P, (mN)) load ramp and resulting penetration depth (h, (nm)) as a function of the time.





(c)  $\Sigma(\Delta h_{\text{pop-in}}) = 25.54 \text{ nm}$      $\Sigma(\Delta h_{\text{pop-out}}) = 25.82 \text{ nm}$







## Table captions

**Table 1.** Chemical composition of the studied stainless steel AISI 301 LN (*wt. %*).

**Table 1**

	<b>C</b>	<b>Cr</b>	<b>Ni</b>	<b>Mn</b>	<b>Si</b>	<b>Mo</b>	<b>N</b>	<b>Fe</b>
<b>AISI 301 LN</b>	0.02	17.48	7.03	1.23	0.45	0.12	0.12	Bal.