

Chapter V :

Mechanisms of

Strain-induced Phase

Transformations in

Fe-Ni-Cr and Fe-Mn-

Al-Si Steel Grades

V.1 Introduction

As it was presented in Chapter I, the quest for steel grades with excellent mechanical properties led to the study of phase transformations in highly alloyed steels. The transformations taking place in austenitic stainless steels are already well known. However, the details of the transformation steps, orientation relationships and variant selection are still not completely elucidated. Moreover, in the

case of high-manganese steels, it is mostly the binary Fe-Mn alloys [Cote95, Jun98, Lee00] and the Fe-Mn-Si or Fe-Mn-Si-Cr shape-memory alloys [MnLR96, Ande98, Jiang98, Chen99, Bliz04] that were investigated. In these cases, the austenite was shown to be metastable and mechanically transform into ϵ -martensite. In Fe-Mn-Si-Al alloys, the formation of α' -martensite has also been reported [Gräs98, From03] and its mechanism of formation deserves a dedicated study. Moreover, the texture is directly related to the mechanisms of phase transformations. This influence is described by two phenomena : the orientation relationships and the variant selection. Understanding these two processes requires a thorough crystallographic study of the phase transformation mechanisms.

The orientation relationships of a phase transformation describe the correspondences between planes and directions of the mother and daughter phases. These correspondences have been deduced from different measurements and therefore several sets of rules have been calculated, as presented in section I.3.1.3. The relationships that will be used in this Chapter are the Burgers relationship for the $\gamma \rightarrow \epsilon$ transformation and the Kurdjumov-Sachs (KS) relationship for the $\gamma \rightarrow \alpha'$ transformation. The Burgers relationship predicts that FCC $\{111\}_{\gamma}$ and HCP $\{0001\}_{\epsilon}$ planes as well as FCC $\langle 1-10 \rangle_{\gamma}$ and HCP $\langle -12-10 \rangle_{\epsilon}$ planes are parallel [Lecr72]. According to the Kurdjumov-Sachs relationship, FCC $\{111\}_{\gamma}$ planes and BCC $\{011\}_{\alpha}$ planes are parallel, as well as FCC $\langle 011 \rangle_{\gamma}$ and BCC $\langle 111 \rangle_{\alpha}$ directions [Gode03]. In the case of the $\epsilon \rightarrow \alpha'$ transformation, the combination

of both relationships shows that BCC $\{011\}_{\alpha}$ and HCP $\{0001\}_{\epsilon}$ planes and BCC $\langle 111 \rangle_{\alpha}$ and HCP $\langle -12-10 \rangle_{\epsilon}$ directions are parallel.

However, among all possible variants that could be formed in one grain according to the prediction of the orientation relationships, it is often observed [Chap90, Gode3] that some are more frequently present than others. This selection of variants depends on two factors : the mechanism of phase transformation and the misorientation between the transformation strain and the local stress field. Indeed, it can be quite easily understood that the most frequent variants are the ones for which the transformation strain is “favourably oriented” with respect to the local stress field.

A deep understanding of the crystallographic mechanisms is required. The objective of this Chapter is thus to investigate the mechanisms of phase transformations at the scale of one grain as a function of the chemical composition and of the stress state. Two model materials (steel 301LN and steel Mn20) were chosen.

V.2 Materials and experimental procedure

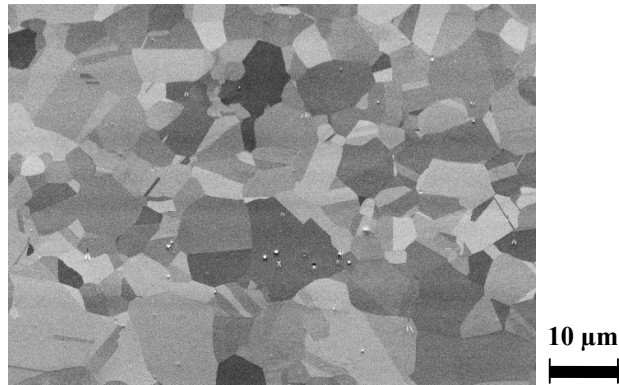
Two steel grades are investigated in this Chapter : steel 301LN, a fully austenitic Ni-Cr stainless steel and steel Mn20, a Fe-Mn-Si-Al alloy. The chemical compositions of these grades are reminded in Table V.1.

*Mechanisms of Strain-induced Phase Transformations
in Austenitic Stainless Steels and Fe-Mn-Si-Al Steels*

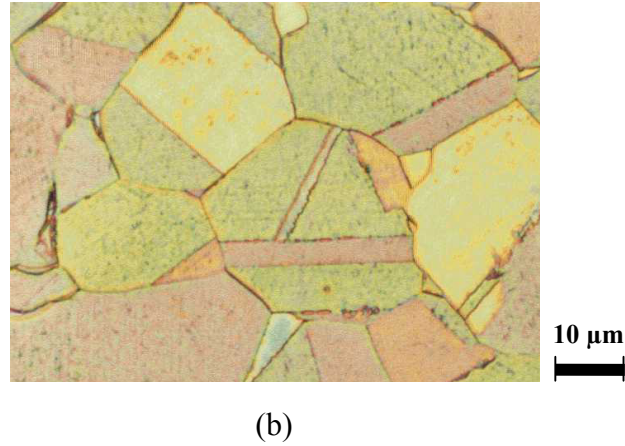
wt. %	C	Mn	Al	Si
301LN	0.027	1.3	< D. L.	0.43
Mn20	$1.7 \cdot 10^{-3}$	19.66	3.11	2.88
wt. %	Cr	Ni	Mo	N
301LN	17.51	6.59	0.16	0.012
Mn20	< D. L.	< D. L.	< 0.005	$5.7 \cdot 10^{-5}$

*Table V.1 : Chemical compositions of the different steel grades.
(D.L. stands for Detection Limit)*

Steel Mn20 was hot-rolled then annealed for 1h at 1000°C. After this annealing treatment, it contained 96% of austenite and 4% of ferrite. The microstructure of steels 301LN and Mn20 1000 are reminded on Figure V.1. The austenite mean grain size are 11 μ m and 22 μ m, respectively.



(a)



*Figure V.1 : Microstructures of steels 301LN (a)
and Mn20 1000 (b) prior to deformation.*

In order to induce the martensitic transformation, the samples of steel Mn20 1000 were strained by uniaxial tension while steel 301LN was deformed by uniaxial tension, cold-rolling or rotary swaging. Deformed specimens were polished and electro-polished before observations by TEM and OIM.

Four phases are found in the microstructure : ferrite and austenite are present before straining while ϵ - and α' -martensite appear during straining. Table V.2 presents the lattice parameters (a and c) of the different phases used in OIM microscopy.

	austenite	Ferrite (& α' -martensite)	ϵ -martensite
<i>a</i>	3.6	2.8	2.54
<i>c</i>	-	-	4.16

Table V.2 : Lattice parameters of the different phases.

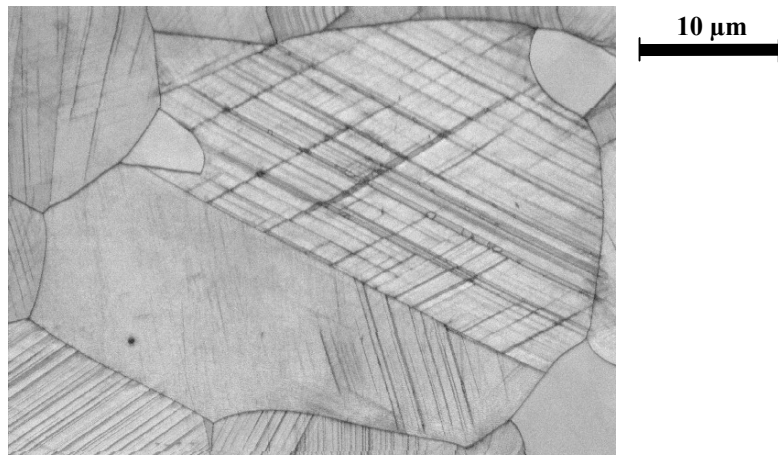
V.3 Results

V.3.1 Strain-induced phase transformations in Fe-Mn-Si-Al steels

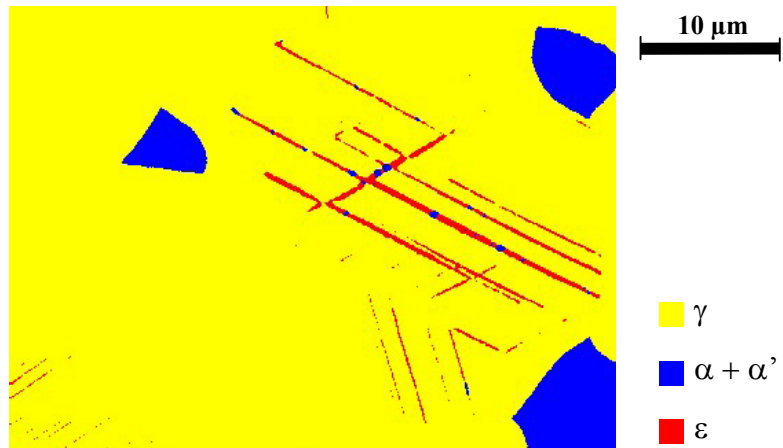
Figure V.2 presents band contrast map, phase map and inverse pole figure (IPF) maps along the rolling direction (which corresponds to the tensile direction) of the microstructure of steel Mn20 1000 after tensile straining to $\epsilon = 0.05$. The band contrast map (Figure V.2 (a)) presents one larger grain that appears to be divided in two and to contain two systems of thin parallel bands in its upper part and one system in its lower part. Figure V.2 (c) shows that these two parts are twin-related. Figure V.2 (b) confirms that the microstructure is mostly austenitic, with three small grains of ferrite surrounding the central austenite grain. Moreover, the intragranular bands are ϵ -martensite with a thickness of around 1 to 2 μm for the thickest ones. These bands are blocked mostly by grain or twin boundaries and sometimes by other ϵ bands. Small grains of α' -martensite can be found within these ϵ bands, especially at the junction of two bands. Figure V.2 (d) shows

that all the small grains of α' -martensite within one particular ε band exhibit the same crystallographic orientation. All the laths of ε -martensite which are parallel also exhibit the same crystallographic orientation (Figure V.2 (e)).

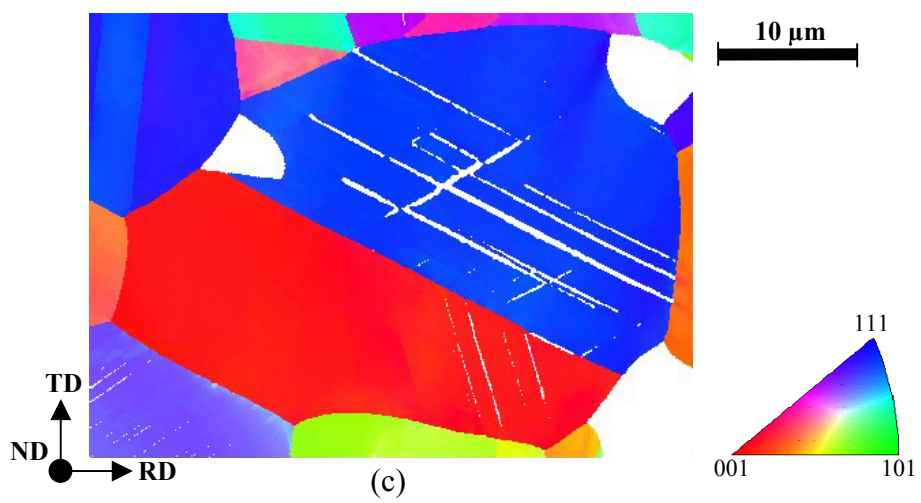
*Mechanisms of Strain-induced Phase Transformations
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(a)



(b)



(c)

*Mechanisms of Strain-induced Phase Transformations
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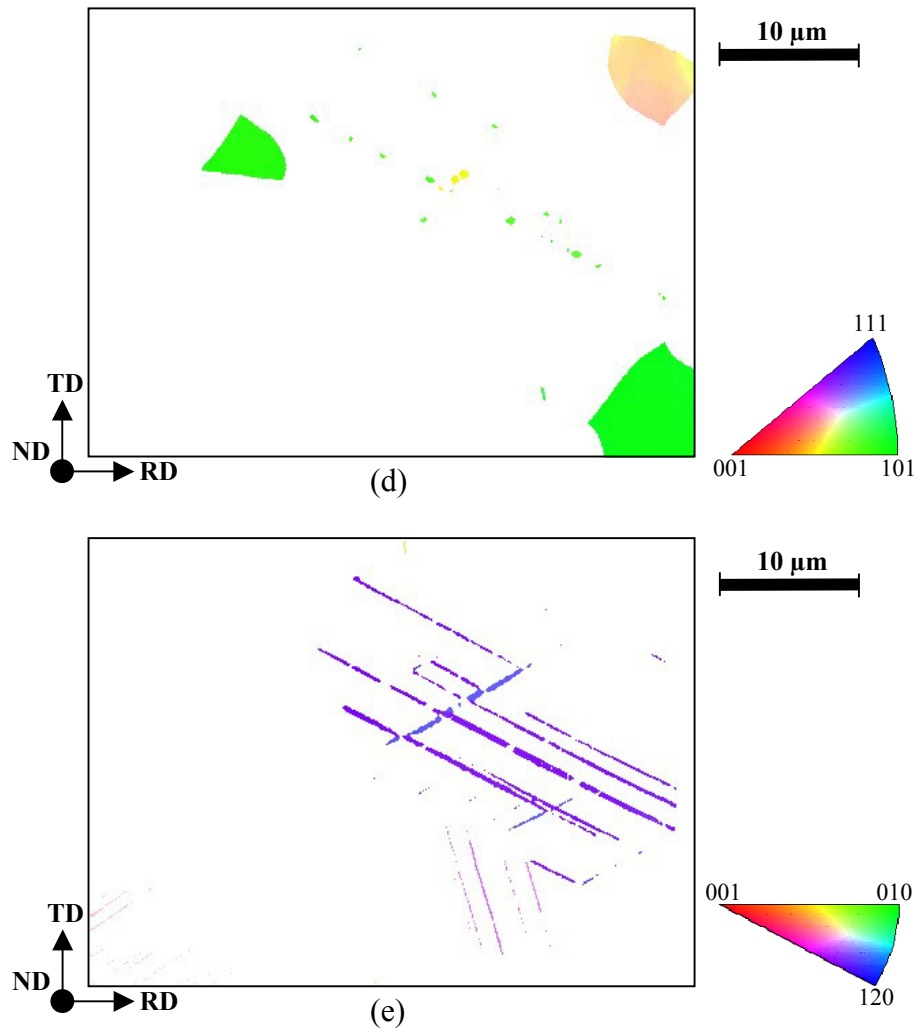


Figure V.2 : EBSD maps of steel Mn20 1000 strained to $\varepsilon = 0.05$:

(a) band contrast ;

(b) phase contrast ;

and IPF maps along the rolling direction (RD) for

(c) the austenite ;

(d) the ferrite and α' -martensite ;

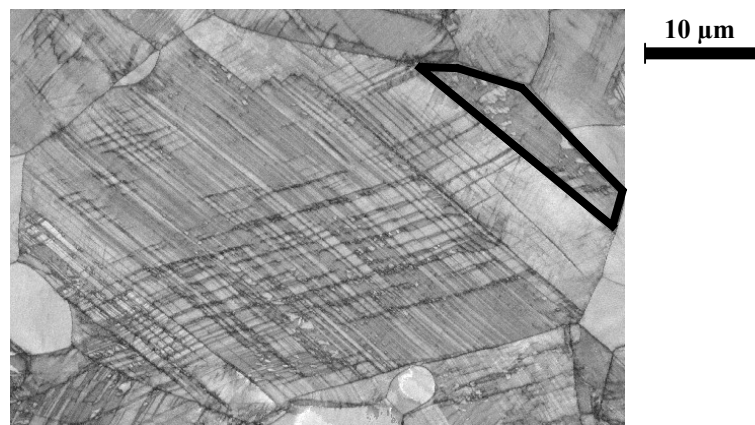
(e) the ε -martensite.

Band contrast, phase and inverse pole figure (IPF) maps of the microstructure of steel Mn20 1000 deformed to $\varepsilon = 0.16$ are presented on Figure V.3. One large central grain can be observed on the band contrast map (Figure V.3 (a)). It contains two systems of thin parallel bands and some of these bands contain smaller grains. The phase map (Figure V.3 (b)) shows that the central grain is austenitic and the surrounding small grains are ferritic grains. The proportions of ε bands and of α' -martensite grains have strongly increased compared to Figure V.2, although these are unequally distributed in the austenite grain. Indeed, the density of ε - and α' -martensite is very large in some parts of the grain (highlighted by the black box) while the transformation has barely started in other areas. Figure V.3 (c) shows that this austenite grain contains two annealing twins. Figure V.3 (d) demonstrates that only two different crystallographic orientations can be observed for the α' grains. Furthermore, these grains are contained within parallel bands of ε -martensite presenting the same lattice orientation, as shown on Figure V.3 (e).

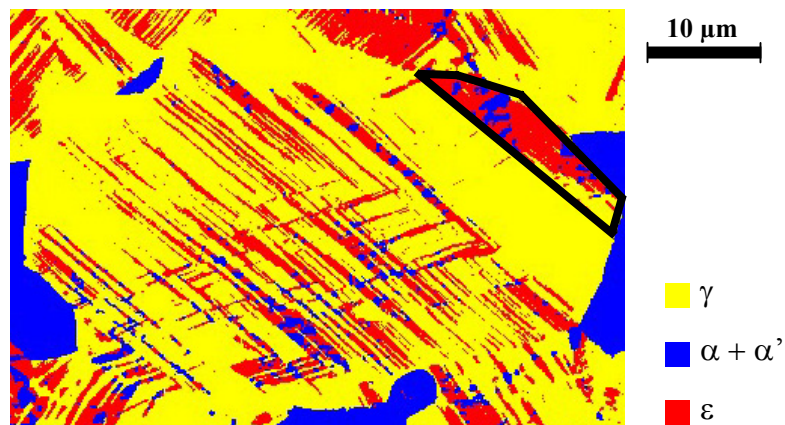
Figure V.4 presents the OIM micrographs of steel Mn20 1000 strained to $\varepsilon = 0.16$. Figure V.4 (a) shows that the central grain exhibits 3 systems of thin bands (named ε_1 , ε_2 and ε_3 by decreasing order of frequency) which contain small grains. The phase map (Figure V.4 (b)) shows that the thin bands are ε -martensite while the small grains within these bands are α' -martensite. The IPF map of austenite and ε -martensite are presented on Figure V.4 (c) and Figure V.4 (d), respectively. A black box highlights a selected part of the grain corresponding to a single crystallographic orientation (no

annealing twins). However, the austenite within this black box presents an important misorientation spread. Three ε orientations (ε_1 , ε_2 and ε_3) can be detected within the austenite grain. This is not a common situation as in most grains, two different orientations of ε -martensite are mostly observed.

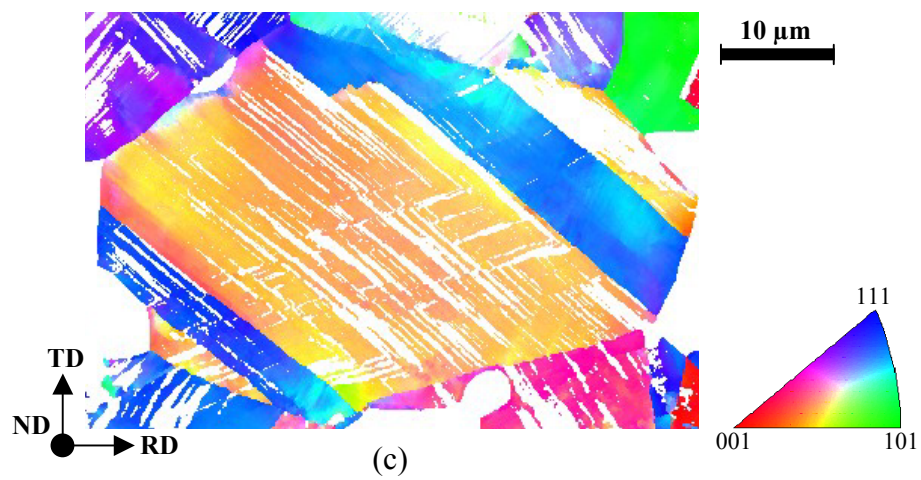
*Mechanisms of Strain-induced Phase Transformations
in Austenitic Stainless Steels and Fe-Mn-Si-Al Steels*



(a)



(b)



(c)

*Mechanisms of Strain-induced Phase Transformations
in Austenitic Stainless Steels and Fe-Mn-Si-Al Steels*

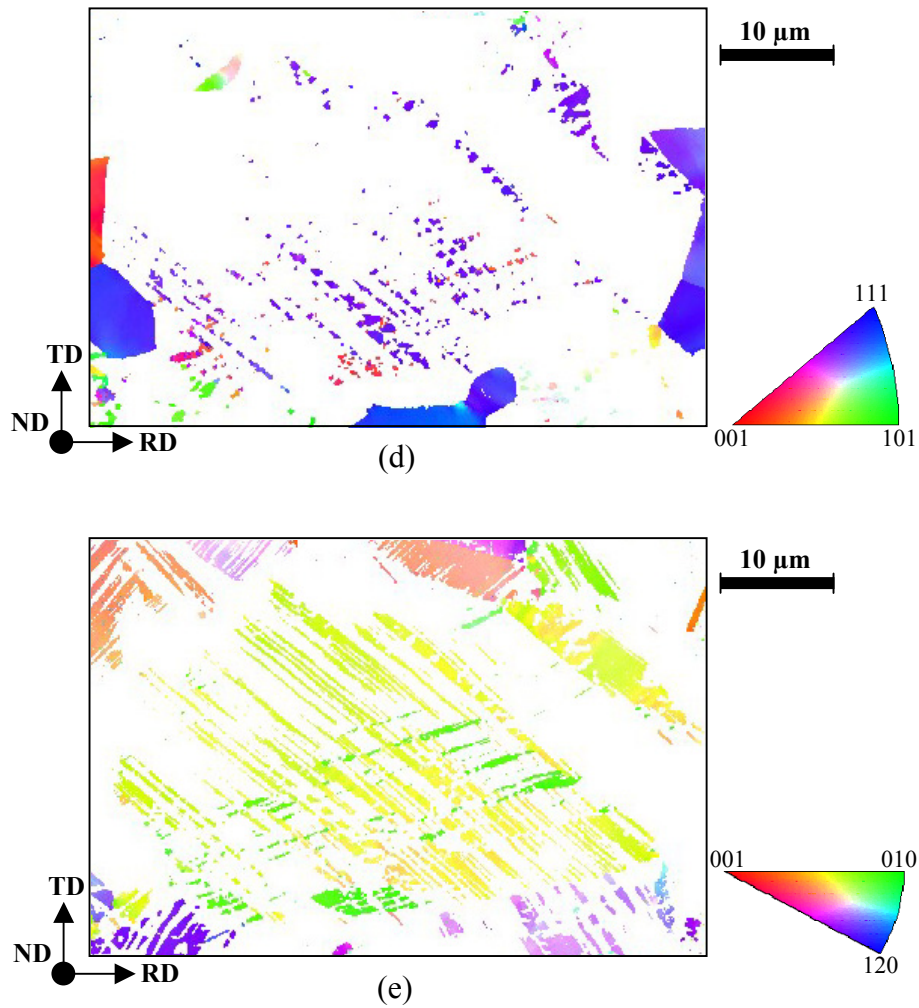
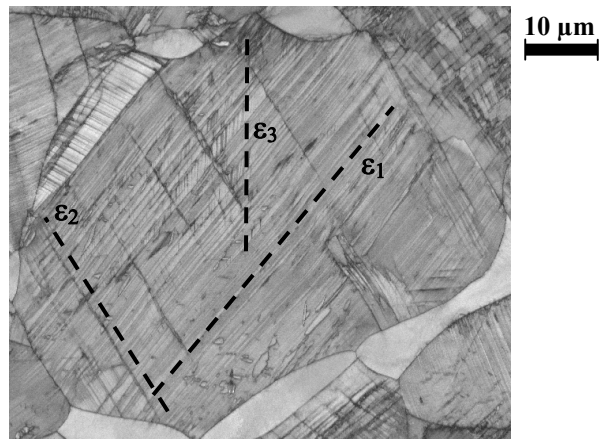


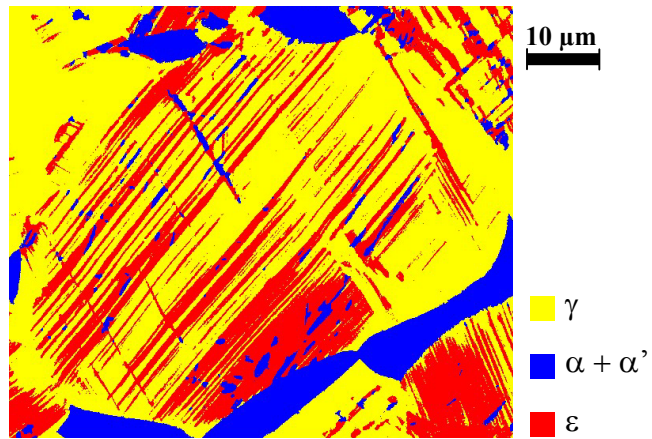
Figure V.3 : EBSD maps of steel Mn20 1000 strained to $\varepsilon = 0.16$:

- (a) band contrast ;*
- (b) phase contrast ;*
- and IPF maps along the rolling direction (RD) for*
 - (c) the austenite ;*
 - (d) the ferrite and α' -martensite ;*
 - (e) the ε -martensite.*

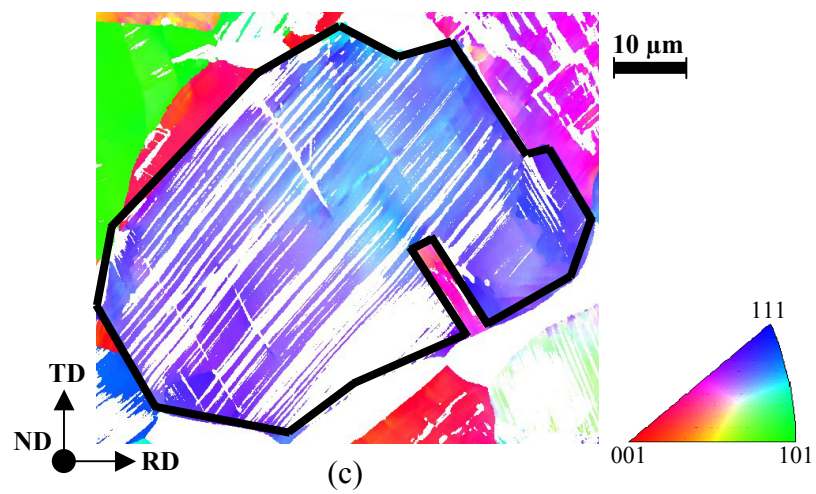
*Mechanisms of Strain-induced Phase Transformations
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(a)



(b)



(c)

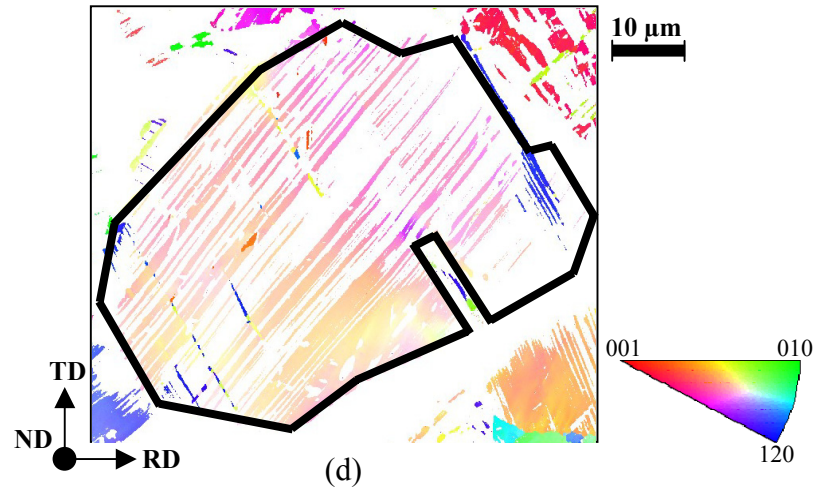


Figure V.4 : EBSD maps of steel Mn20 1000 strained to $\varepsilon = 0.16$:

(a) band contrast with three systems of bands (ε_1 , ε_2 and ε_3) ;

(b) phase contrast ;

and IPF maps along the rolling direction (RD) for

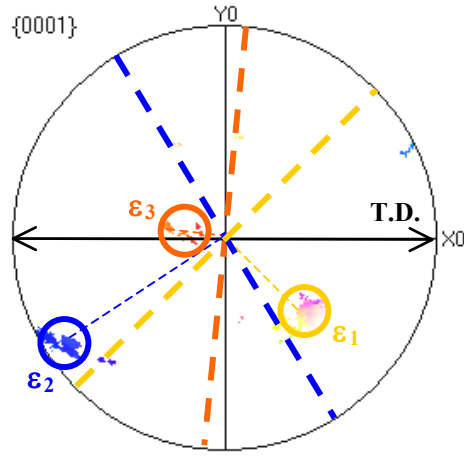
(c) the austenite ;

(d) the ε -martensite.

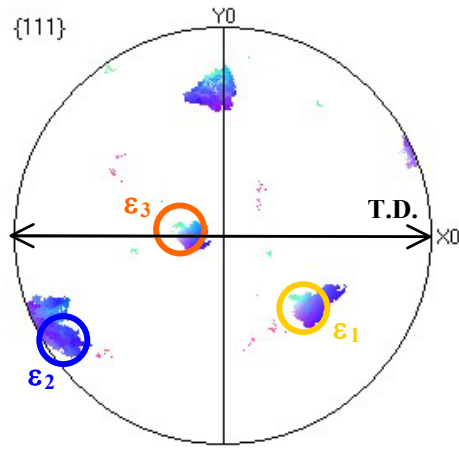
The black box highlights a zone of the austenite grain presenting a single crystallographic orientation (no annealing twins).

Figure V.5 shows the pole figure of the ε -martensite (Figure V.5 (a)) and of the austenite Figure V.5 (b) included in the highlighted zone of Figure V.4. Each pole of ε -martensite corresponds to one ε variant. The pole of each ε variant is superimposed on one pole of the austenite. Moreover, the trace of the ε variants are represented on the pole figure and the angles between these traces and the tensile direction (aligned with the rolling direction) are of 46° , 63° and 84° , respectively. It can be noticed that the trace of the three

variants are exactly parallel with the ϵ bands on the OIM micrograph (Figure V.4 (a)).



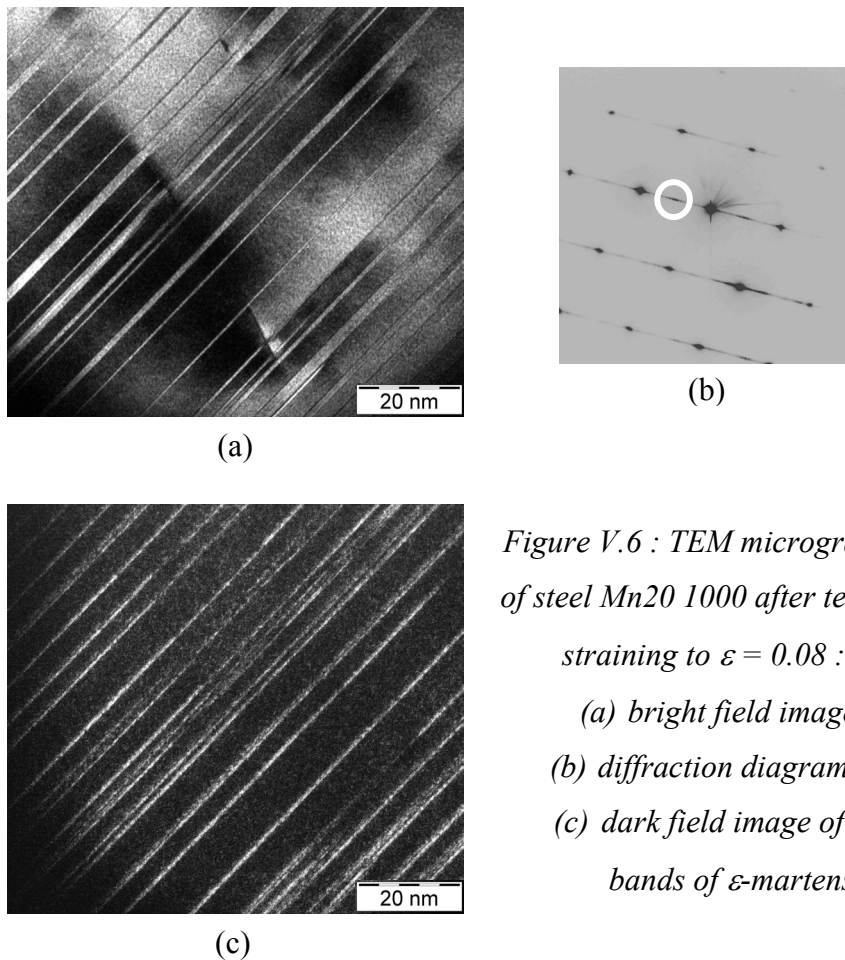
(a)



(b)

Figure V.5 : Pole figures of the ϵ -martensite (a) and austenite (b) within the highlighted area in Figure V.4. The traces of the planes are represented on the pole figure for the ϵ -martensite.

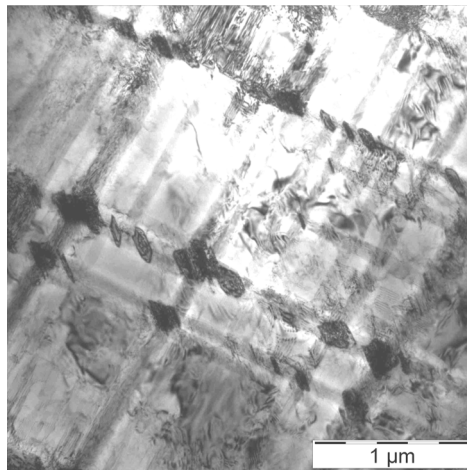
Figures V.6, V.7 and V.8 present TEM micrographs¹ of steel Mn20 1000 strained to $\varepsilon = 0.08$. Figure V.6 (a) shows that bands with a thickness of a few nanometres are present in the material. The diffraction pattern of Figure V.6 (b) contains streaks corresponding to the ε phase visible between the austenite spots. The dark field image of these ε bands is given on Figure V.6 (c).



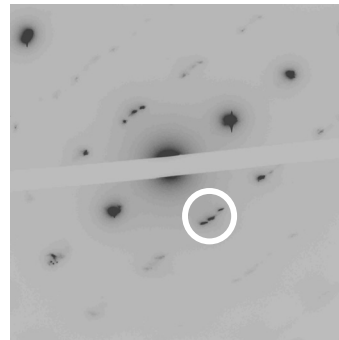
*Figure V.6 : TEM micrographs
of steel Mn20 1000 after tensile
straining to $\varepsilon = 0.08$:
(a) bright field image ;
(b) diffraction diagram and
(c) dark field image of thin
bands of ε -martensite.*

¹ The smallest objective aperture of the TEM was $30\mu\text{m}$, corresponding to a selection diameter of $0.4\text{ \AA}^{-1}$ on the diffraction pattern. Therefore, the spot selection for the dark field images is not enough accurate to observe the orientation relationships between the phases.

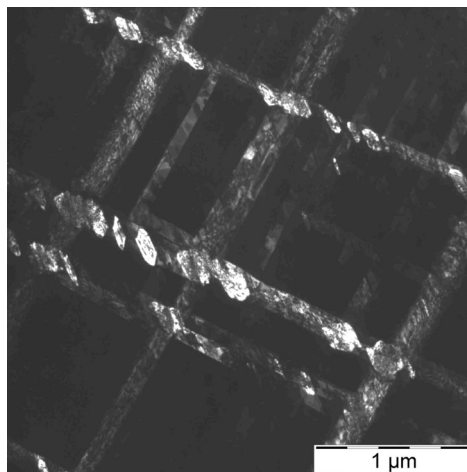
Figure V.7 illustrates another configuration of the ϵ bands and the presence of α' grains. Bright field image (Figure V.7 (a)) shows two systems of bands with small grains at their intersections. On the diffraction diagram (Figure V.7 (b)), spots corresponding to the α' phase can be observed besides the spots of the austenite mother phase. The α' -martensite grains appear at the intersection of two bands on dark field micrographs (Figure V.7 (c)).



(a)



(b)



(c)

*Figure V.7 : TEM micrographs
of steel Mn20 1000 after
tensile straining to $\epsilon = 0.08$:
bright field image (a),
diffraction diagram (b) and
dark field image (c) of the α' -
martensite grains at the
intersection of two ϵ bands.*

Figure V.8 shows that small austenite grains with a different orientation can also be found at the intersection of ϵ -martensite bands. Two systems of bands can be observed on Figure V.8 (a). The diffraction diagram of Figure V.8 (b) shows streaks corresponding to the ϵ phase. Finally, the dark field images of Figure V.8 (c) and Figure V.8 (d) correspond to the two systems of ϵ bands.

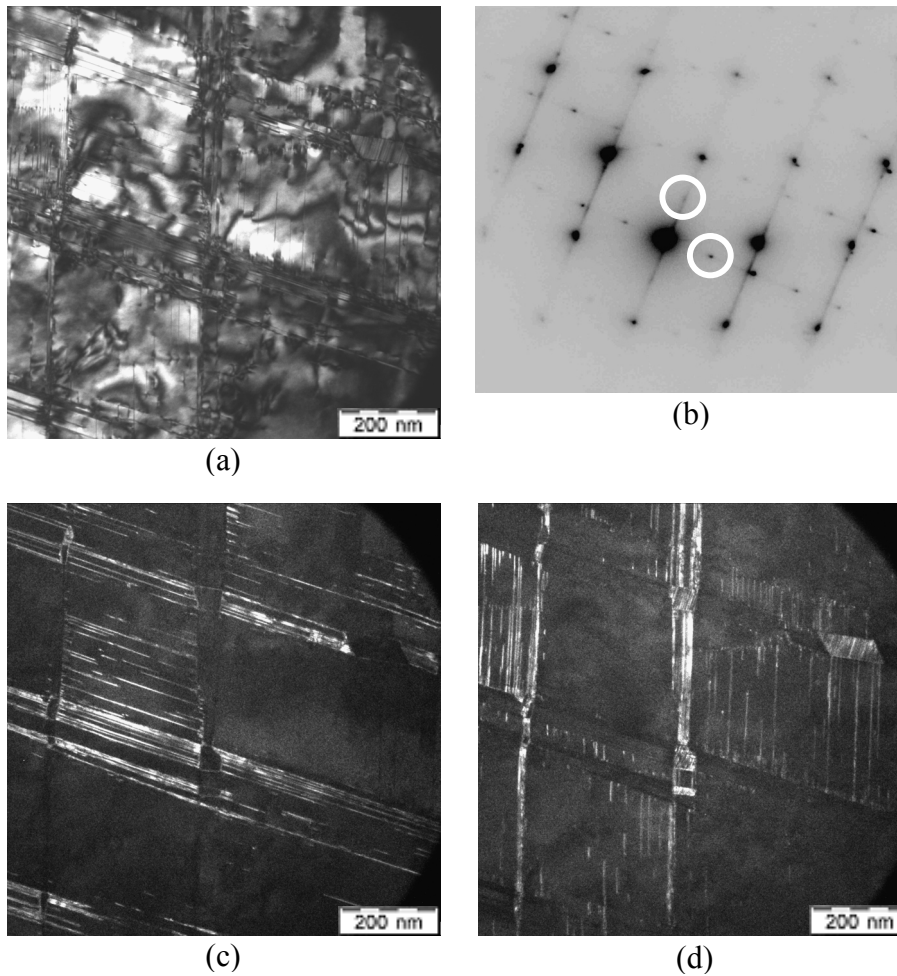


Figure V.8 : TEM micrographs of steel Mn20 1000 after tensile straining to $\epsilon = 0.08$: (a) bright field image, (b) diffraction diagram, (c) and (d) dark field images of both systems of ϵ bands.

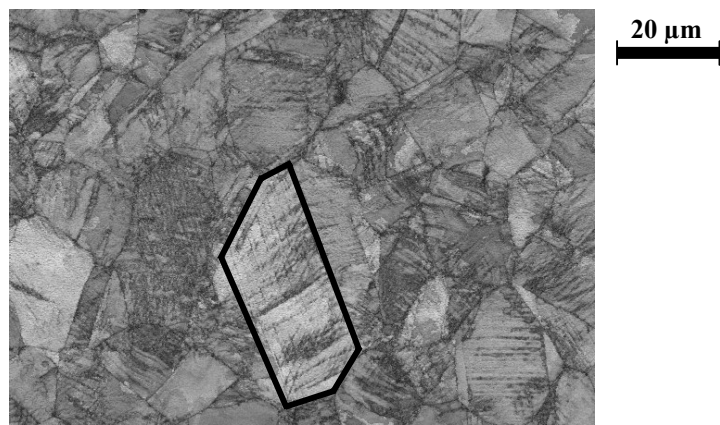
V.3.2 Strain-induced phase transformations in Fe-Ni-Cr stainless steels

Figure V.9 (a) shows an OIM band contrast map of steel 301LN after cold rolling to $\varepsilon = 0.16$. Several systems of thin bands and smaller grains appear within all the grains. It appears on Figure V.9 (b) that the proportion of ε -martensite is very low. Moreover, some austenite grains are almost completely transformed into α' -martensite. In less transformed grains (such as the grain highlighted by the black box), the proportion of ε -martensite is higher. In these grains, it appears that the α' -martensite is found preferentially along the ε bands, but they do not stay restricted within these bands. The IPF maps along the rolling direction (which corresponds to the tensile direction) for the austenite, α' - and ε -martensite are presented on Figure V.9 (c), (d) and (e), respectively. It can be seen on Figure V.9 (c) that some austenite grains present annealing twins. It is also worth noticing that the untransformed austenite grains exhibit misorientation spreads. Figure V.9 (d) shows that the α' -martensite grains present several orientations in each austenite grain.

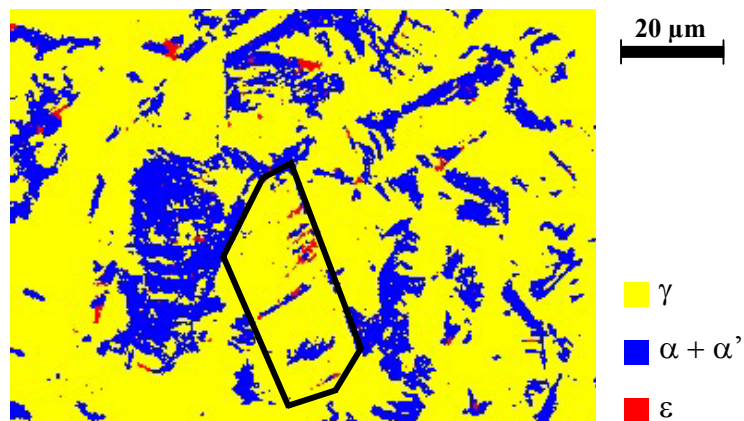
Figure V.10 presents OIM micrographs of the microstructure of steel 301LN after rotary swaging to $\varepsilon = 0.12$. Figure V.10 (a) presents a band contrast map showing one or two systems of thin bands within the grains. No ε phase can be found on Figure V.10 (b). These bands appear to be composed of austenite and α' -martensite. Small grains of α' -martensite have also formed along the grain boundaries or along

the bands. Various orientations of α' -martensite are found to appear on one system of parallel bands (Figure V.10 (d)). Due to the symmetry of the sample and of the applied strains, the sample coordinate system is characterised by the swaging direction (SD) and two axis X and Y, defining the plane which is orthogonal to SD.

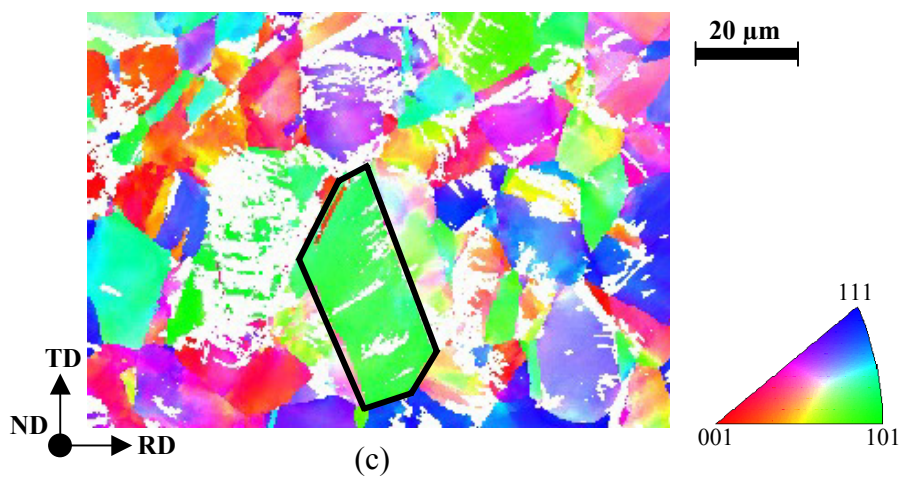
*Mechanisms of Strain-induced Phase Transformations
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(a)



(b)



(c)

*Mechanisms of Strain-induced Phase Transformations
in Austenitic Stainless Steels and Fe-Mn-Si-Al Steels*

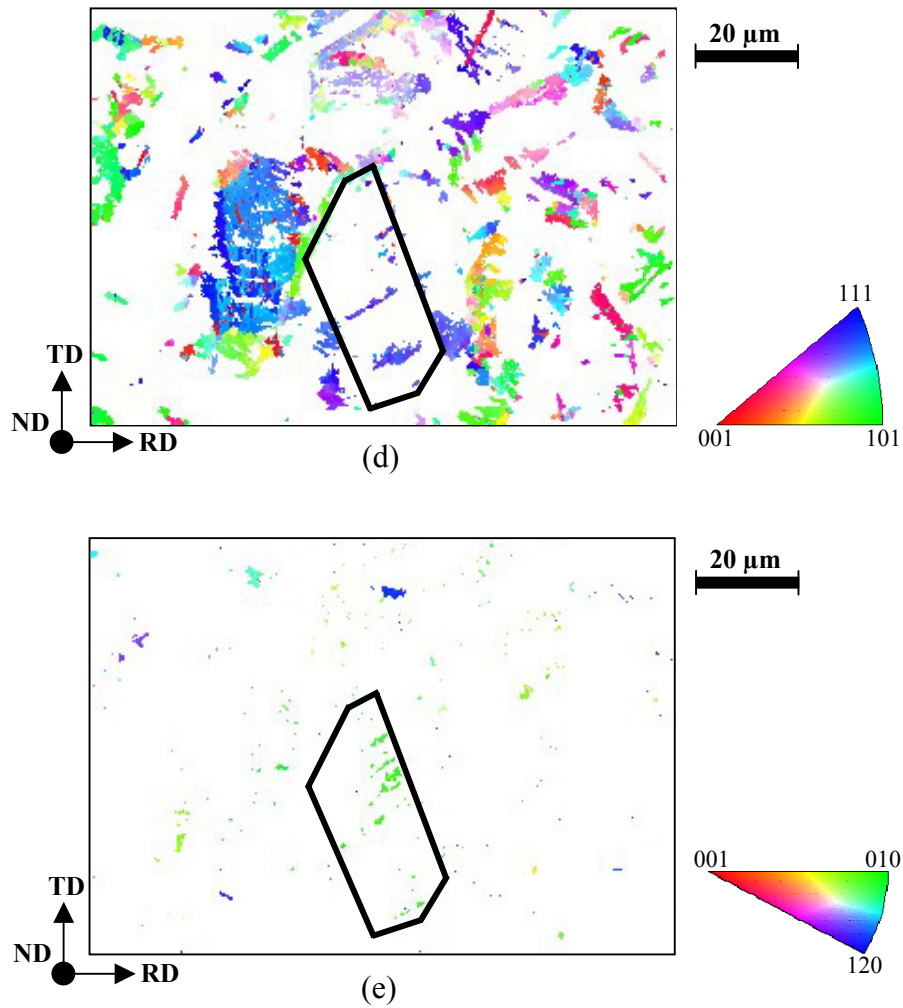
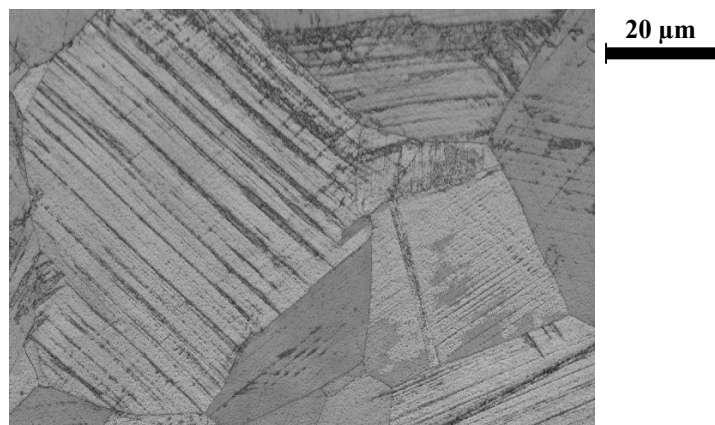


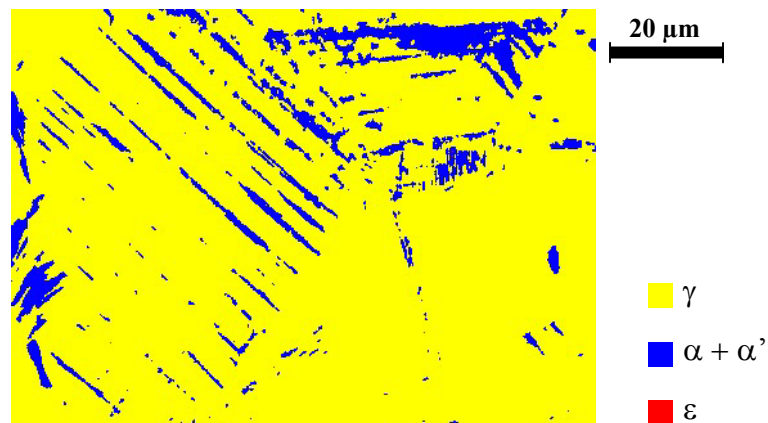
Figure V.9 : EBSD maps of steel 301LN cold-rolled to $\varepsilon = 0.16$:

- (a) band contrast ;*
- (b) phase contrast ;*
- and IPF maps along the rolling direction (RD) for*
 - (c) the austenite ;*
 - (d) the ferrite and α' -martensite ;*
 - (e) the ε -martensite.*

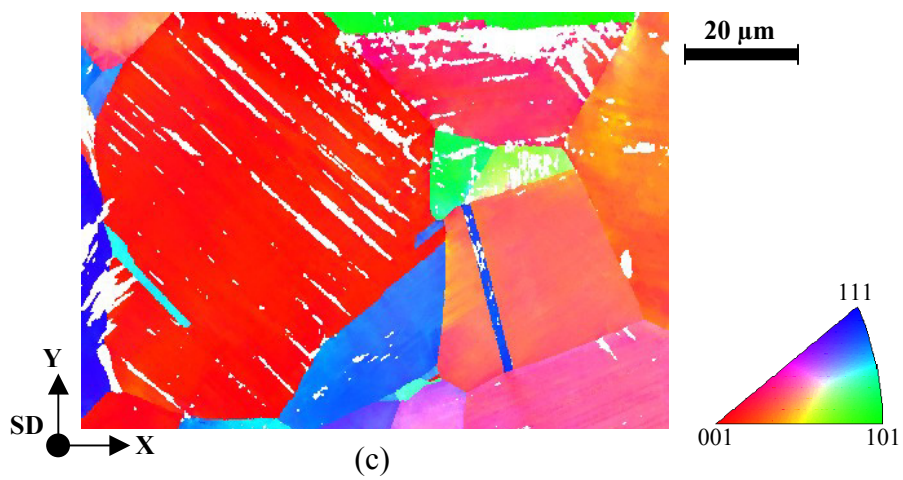
*Mechanisms of Strain-induced Phase Transformations
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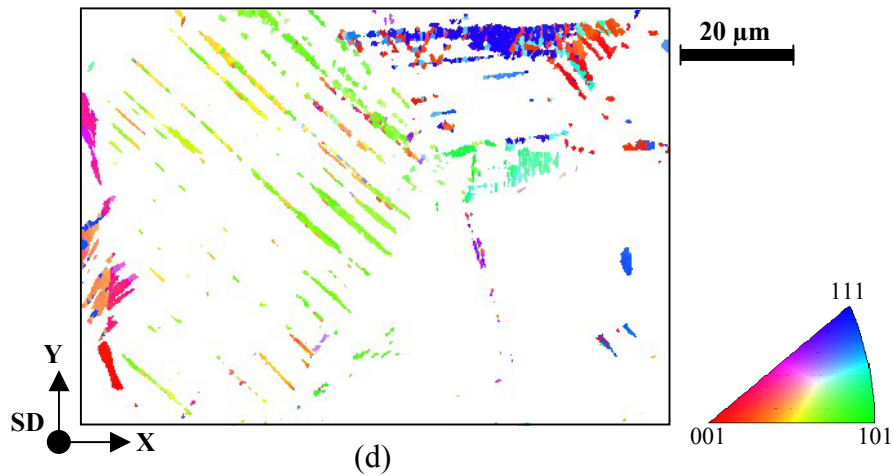
(a)



(b)



(c)



*Figure V.10 : EBSD maps of steel 301LN strained
by rotary swaging to $\varepsilon = 0.12$:*

(a) band contrast ;

(b) phase contrast ;

and IPF maps along the swaging direction (SD) for

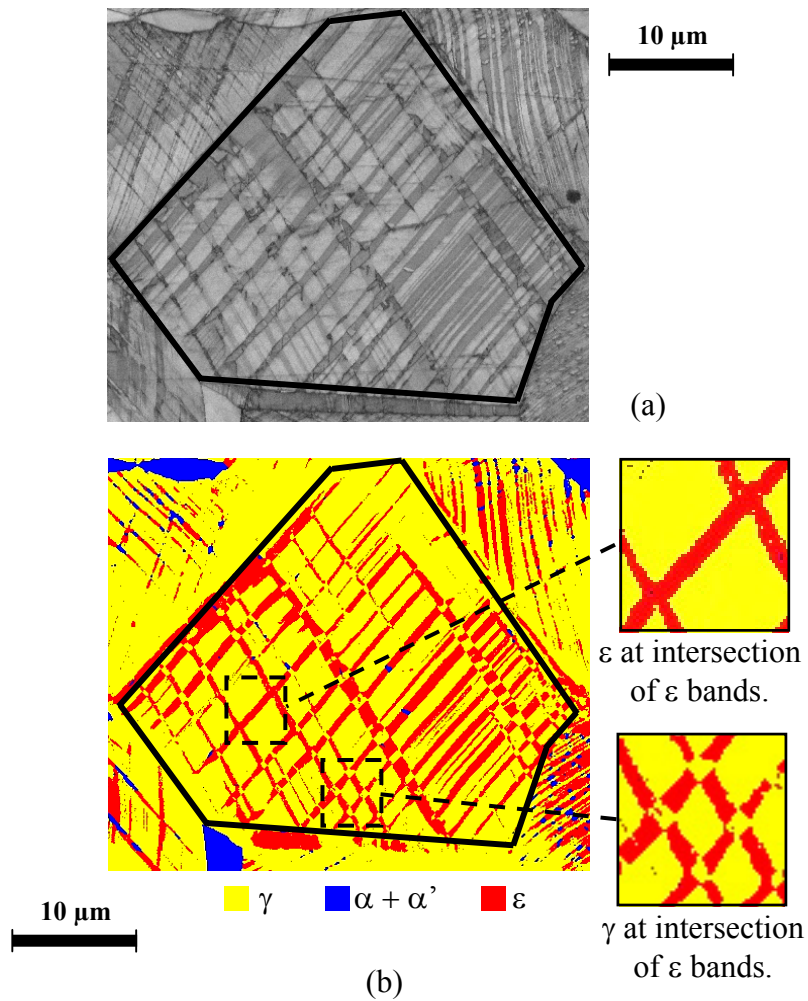
(c) the austenite ;

(d) the α' -martensite.

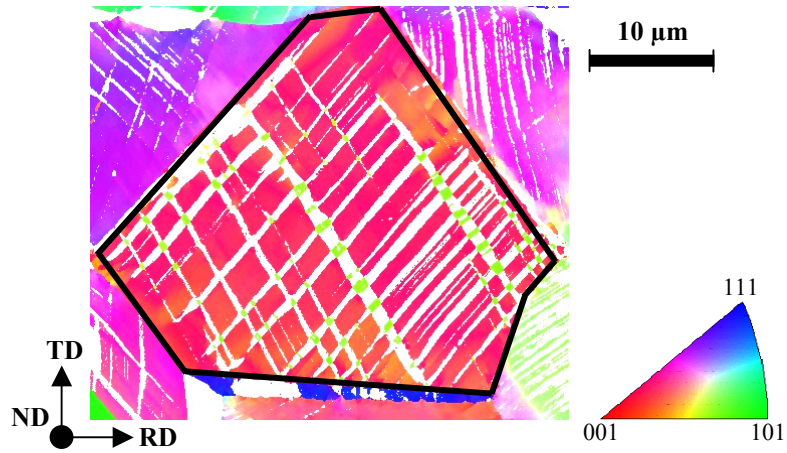
V.3.3 Particular cases of phase transformations in Fe-Mn-Si-Al steels

At the intersection of two bands of ε -martensite, grains of austenite and ε -martensite have been observed, although this phenomenon is not very common. Figure V.11 presents OIM micrographs of the microstructure of steel Mn20 1000 after tensile straining of $\varepsilon = 0.12$. Figure V.11 (a) presents the band contrast map

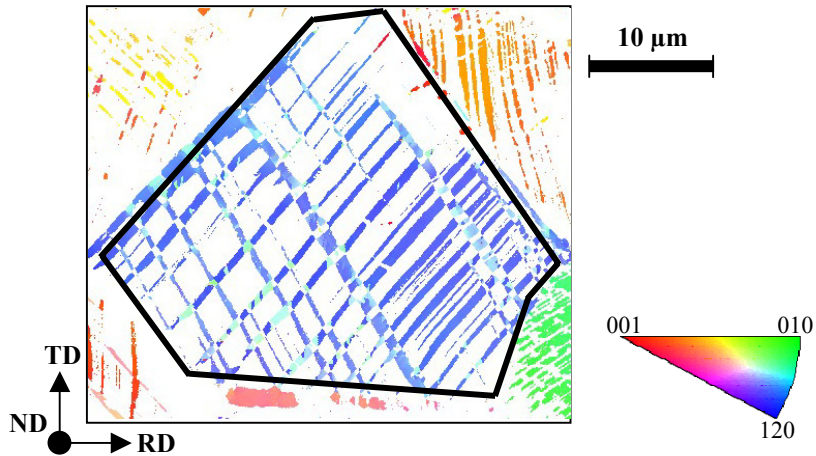
of one austenite grain (surrounded by a black box) containing two systems of thin bands. Small grains are visible within these bands, mostly at the intersection of two bands. As shown by the phase map of Figure V.11 (b), the bands are composed of ϵ -martensite, while their intersections may be made of austenite or ϵ -martensite. Moreover, Figure V.11 (c) shows that the orientation of these small austenite grains differ from the orientation of the mother austenite. Finally, Figure V.11 (d) shows that some parts of the ϵ bands exhibit a different orientation with respect to the rest of the band.



*Mechanisms of Strain-induced Phase Transformations
in Austenitic Stainless Steels and Fe-Mn-Si-Al Steels*



(c)



(d)

Figure V.11 : EBSD maps of steel Mn20 1000

tensile strained to $\varepsilon = 0.12$:

(a) band contrast and (b) phase map ;

and IPF maps along the rolling direction (RD) for :

(c) austenite and (d) ϵ' -martensite.

For a better understanding, Figure V.12 presents the superimposition of the IPF maps of Figure V.11 (c) and (d). A misorientation analysis shows that these zones of secondary ε phase are “twin-related” to the primary ε bands (corresponding to a rotation of about 87° around a $\langle 1-210 \rangle$ axis with a $\{10-12\}$ twinning plane). The secondary ε zones are located at the intersection of two primary ε bands or next to it when the intersection consists in a secondary austenite grain.

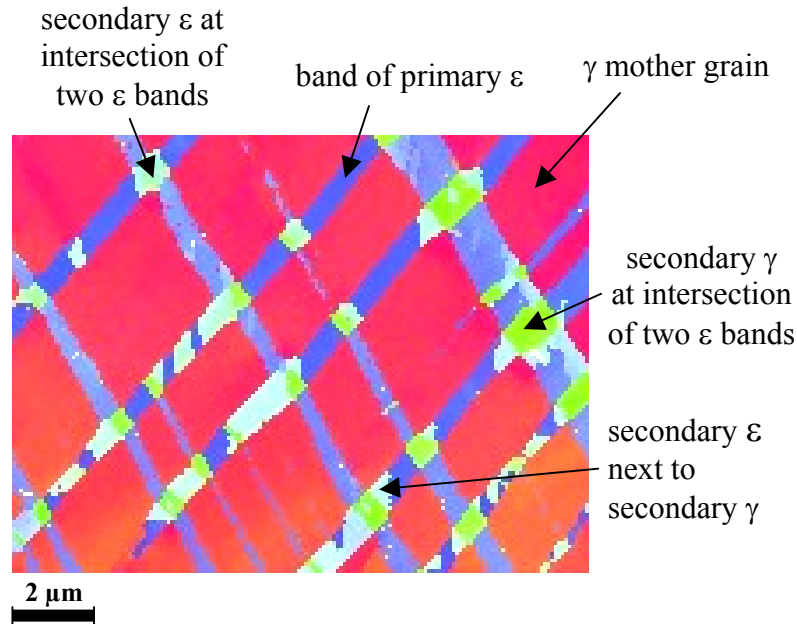


Figure V.12 : Superimposition of IPF maps along the rolling direction (RD) for the austenite and the ε -martensite in Mn20 1000 tensile strained to $\varepsilon = 0.12$.

Figure V.13 shows the pole figure, in the coordinate system of the austenite, of the austenite (Figure V.13 (a)) and ε -martensite

(Figure V.13 b)) corresponding to the highlighted grain (black box) of Figure V.11. The pole figure of ϵ phase shows that to each the two primary ϵ variants (poles surrounded in black) corresponds a twinned fraction (poles surrounded in red). The austenite pole figure shows that if the mother grain has one pole in common with each of the ϵ variants, the secondary γ grains have one pole in common with each of the secondary ϵ variants. Moreover, for both phases, a misorientation spread can be found and the poles of the primary and secondary variants are in contact.

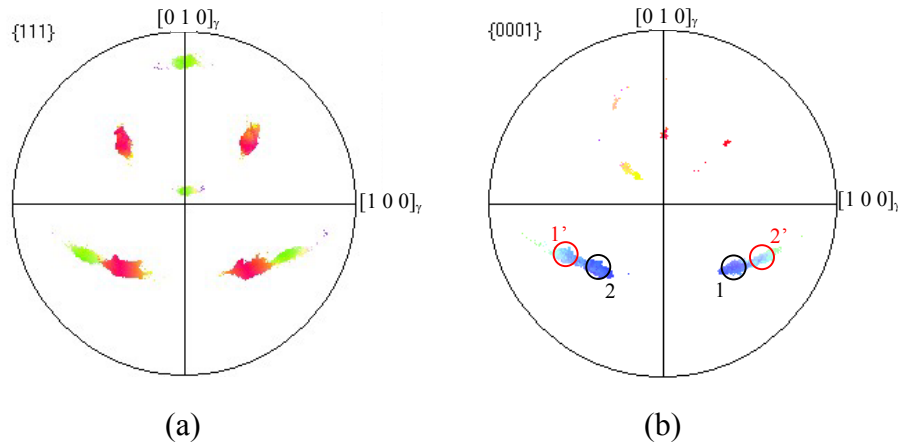


Figure V.13 : Pole figures of austenite (a) and ϵ -martensite (b) corresponding to the highlighted grain (black box) of Figure V.11.

V.4 Discussion

It has been shown in the literature that the mechanically-induced phase transformations in metastable austenitic stainless steels bring

about the formation of ϵ - and α' -martensite. Furthermore, the ϵ -martensite acts as an intermediary phase in the formation of α' -martensite [Good70, Broo79a, Broo79b, Lee01a, Lee01b]. These successive transformations have been observed in steels 301LN and Mn20. Moreover, the austenite seems to transform also directly into α' -martensite in steel 301LN.

V.4.1 Mechanisms of strain-induced phase transformations

V.4.1.1 Transformation of austenite into ϵ -martensite

As it can be observed in Figures V.3 and V.4, the first transformation to take place in the austenite grains of steel Mn20 brings about thin bands of ϵ -martensite that cross the austenite grains. The thickness of the ϵ bands observed vary from a few nanometers to a few micrometers. These laths of ϵ -martensite form by the piling up of stacking faults, such as presented in section I.3. According to the Burgers orientation relationships, 4 ϵ variants could form as the stacking faults may appear on the 4 different $\{1\ 1\ 1\}$ close-packed planes. In strain-induced phase transformations, only some of the possible variants are effectively formed. Indeed, the dislocation gliding planes activated by the applied stress determine which variant will appear. In a first step, plastic straining induces the movement of dislocations once the resolved shear stress reaches the critical resolved shear stress (CRSS). The dislocations glide on $\{1\ 1\ 1\}$ planes and the low stacking fault energy (SFE) brings about the dissociation of the

dislocations into partials. Between two partial dislocations, a stacking fault is formed as it is presented in section I.2.3.1. Thin bands of ϵ phase result from several stacking faults packed together [Broo79a, Broo79b]. Therefore, the orientation of the ϵ laths depend on the active gliding planes for the partial dislocations. Depending on the applied external stress and on its relative orientation with respect to the grain lattice orientation, one or several gliding planes will be activated. The planes with the most favourable orientation relatively to the applied stress exhibit the largest resolved shear stress. The transformation into ϵ -martensite would thus take place preferentially on these planes. This is confirmed by Figures V.4 and V.5. Indeed, it appears that the ϵ bands are more frequent on the $\{1\ 1\ 1\}$ planes that are favourably oriented with respect to the applied stress. This corresponds to what has been observed in the literature [Han04, Gey05]. The same mechanism is probably at the origin of the ϵ -martensite formation in steel 301LN. However, this could not be proved as the proportion of ϵ phase is lower and the bands are thinner than in steel Mn20 (which do not allow proper indexation).

V.4.1.2 Formation of α' -martensite

The α' -martensite nucleates within the bands of ϵ -martensite and the most common nucleation site is the intersection of two ϵ bands. The formation of α' -martensite at the intersection of two ϵ variants has been explained analytically by Lacroisey and Pineau [Lacr72]. They showed that the atomic rearrangement taking place at the intersection of two bands of ϵ -martensite can bring about the

formation of a nucleus of α' -martensite, with an orientation relationship with respect to the austenitic phase that is close to the Kurdjumov-Sachs relationships. In steel Mn20, the α' grains were found to appear mostly at the intersections of two ε bands. In stainless steel 301LN, the nucleation of α' grains could not be observed in the present work. However, precedent results in the literature show that in steel 304, the nucleation took place within ε bands but not specifically at their junctions [Gey05]. Figure V.14 (from [Spen04]) shows TEM micrographs of the nucleation of α' -martensite at ε bands crossing in stainless steel 304 (Figure V.14 (a)) or at the intersection of a slip system with a band of ε -martensite (Figure V.14 (b)).

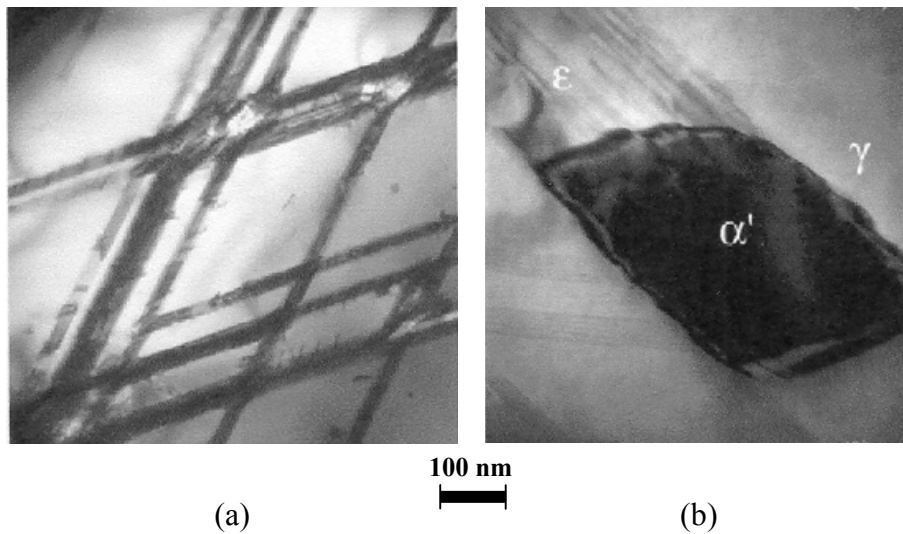


Figure V.14 : TEM micrographs (from [Spen04]) of stainless steel 304 after tensile straining to $\varepsilon = 0.05$ at -198°C :

(a) formation of α' embryos (white) at the intersection of ε -martensite bands and (b) high magnification micrograph of an α' embryo on a band of ε -martensite, with an intersecting slip system (on the left).

The α' nuclei grow up within the band of ε -martensite or within the parent austenite when the plastic deformation increases [Lecr72]. The ε -martensite proportion thus decreases as it transforms into α' -martensite, in stainless steels [Spen04] as well as in high-manganese steels [Oh95]. This mechanism can be observed in both materials in this study. However, a fundamental difference seems to exist between Fe-Mn-Si-Al and stainless steels as it is believed that some direct $\gamma \rightarrow \alpha'$ transformation also took place in steel 301LN only. This direct $\gamma \rightarrow \alpha'$ transformation has been observed in steel 304 by Gey *et al.* [Gey05]. On the other hand, the α' grains grow strictly within the ε bands in steel Mn20 and no direct $\gamma \rightarrow \alpha'$ transformation has been observed.

During rotary swaging of stainless steels, the austenite seems to transform directly into α' -martensite, without visible trace of ε phase. This is due to a quick rise of the sample temperature observed during swaging. This temperature increase combined with the different stress state modifies the relative stability of the phases and the stacking fault energy. However, the absence of ε -martensite in the microstructure is not decisive. Indeed, the increase of temperature could reduce the ε phase stability and induce complete transformation of this phase at the end of each swaging step, when the temperature reaches its maximum. Moreover, the thin bands observed on Figure V.10 could be either ε -martensite too thin to be indexed, shear bands or thin mechanical twins. Lecroisey and Pineau demonstrated that the intersection of two twins constitutes a suitable site for the α' -martensite nucleation [Lecr72] and it was later observed in high-manganese alloys by Oh *et*

al. [Oh95]. Furthermore, during cold drawing of steel 304, Choi and Jin [Choi97] observed the nucleation of α' -martensite grains at the intersection of mechanical twins instead of ε bands. In this case, the high drawing rate is responsible of the temperature rise, inducing a stacking fault energy increase and a modification of the transformation mechanism. The α' -martensite nucleates then mainly at the intersection of mechanical twins. However, in the absence of TEM evidence, the precise mechanism bringing about α' -martensite during rotary swaging could not be determined.

V.4.2 Crystallographic aspects of strain-induced phase transformations

During strain-induced phase transformations, the orientations of the ε - or α' -martensite differ from the predictions of the orientation relationships. Indeed, only some of the predicted variants are actually observed in the strained specimen.

Figure V.15 presents the $\{1\ 1\ 0\}$ poles of α' variants predicted by the KS relationship in the $\{1\ 1\ 1\}$ pole figure of the austenite². The black circles highlight the position of the 4 $\{1\ 1\ 1\}$ poles of the austenite. Following the Burgers orientation relationships, the $\{0\ 0\ 0\ 1\}$ pole of one ε variant (among the 4 possible variants) would superimpose on one of these spots. Following the KS relationships, six $\{1\ 1\ 0\}$ poles of 6 different α' variants (one pole per variant) are

² This figure was made by Prof. S. Godet and graciously offered as a support for the discussion.

superimposed on each of these spots, among the 24 possible variants. All poles of these 6 variants present the same colour (*i.e.* red, green, blue or black) and one particular symbol has been attributed to each variant. Finally, the grey circles surround the superimposition of 2 poles of α' variants presenting a twin relationship with each other. The misorientation between these variants and a variant with a pole on the closest stacking of six poles is thus either 10° or 50° .

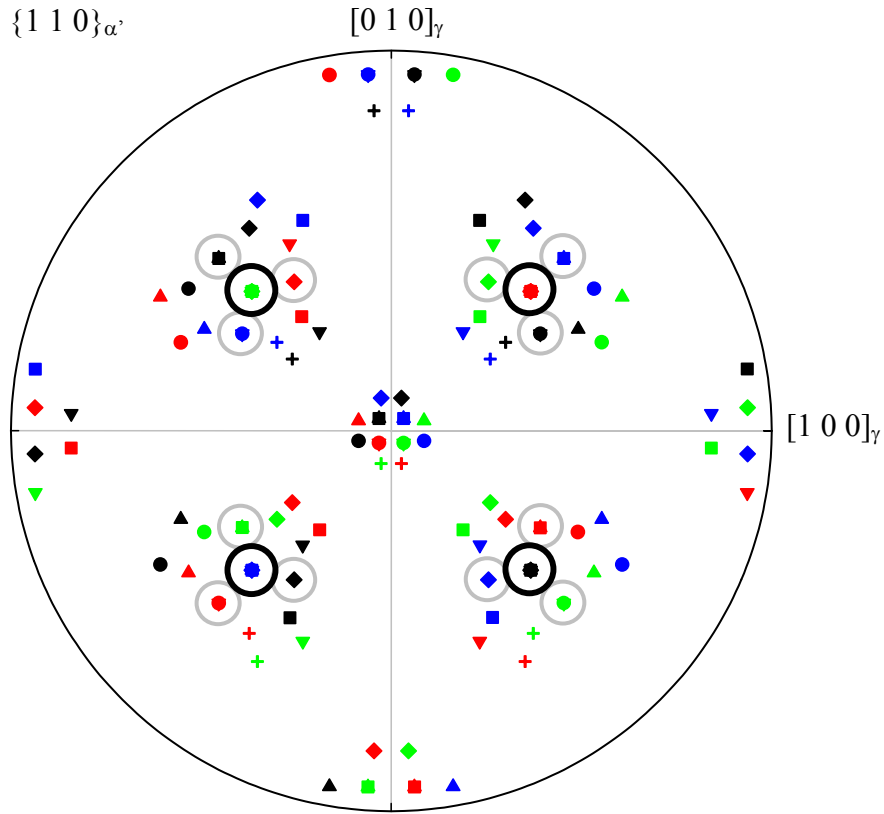
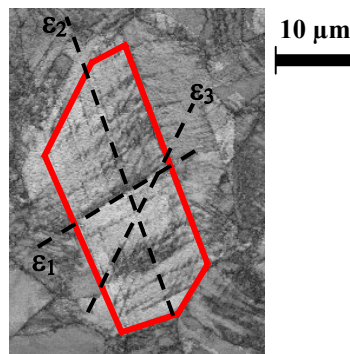


Figure V.15 : Position of the $\{1\ 1\ 0\}$ poles of all the α' variants predicted by the Kurdjumov-Sachs (KS) relationships on a $\{1\ 1\ 1\}$ austenitic pole figure. 6 different α' variants have one pole surrounded by a black circle and 2 surrounded by a grey circle.

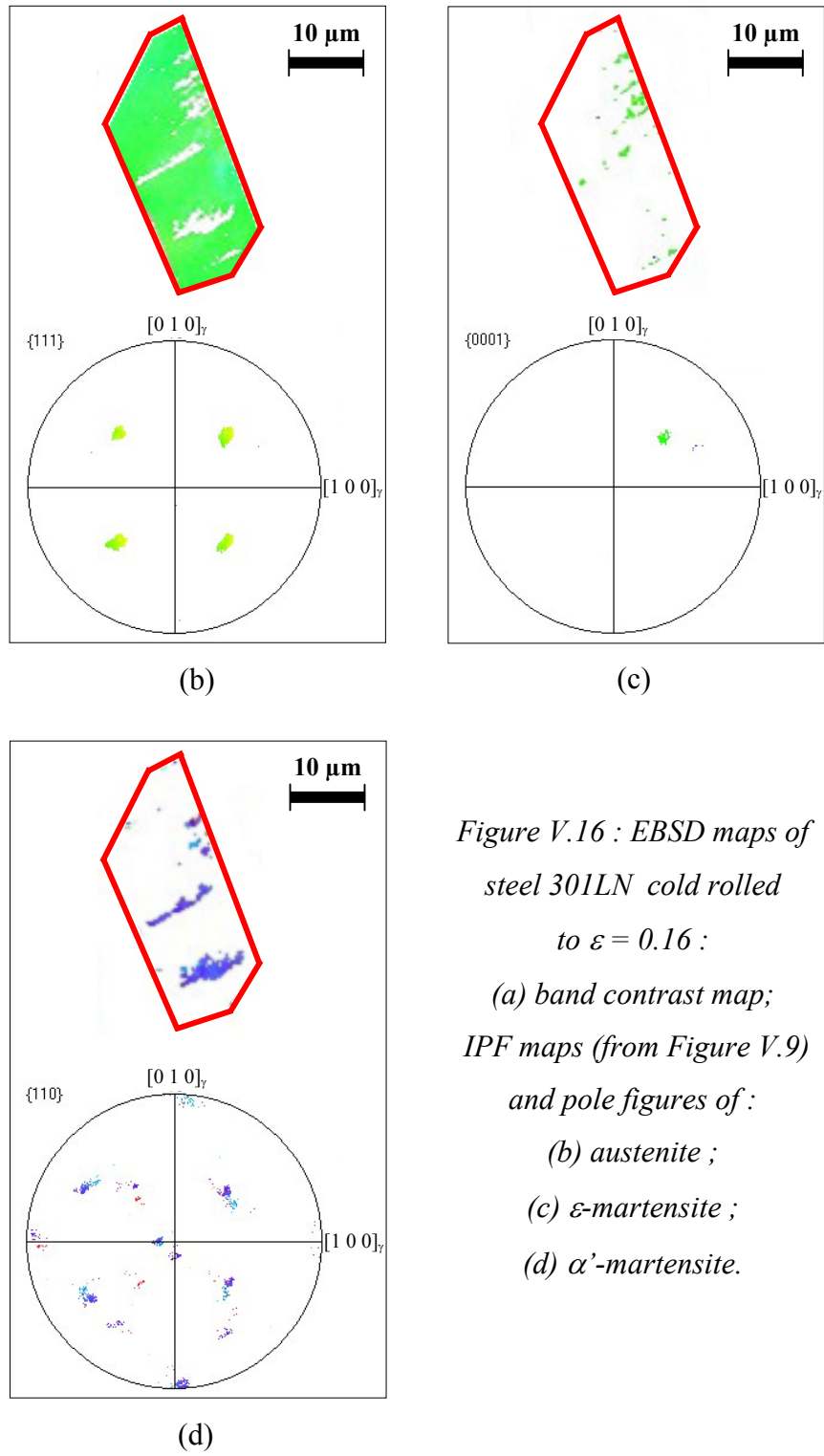
V.4.2.1 Variant selection during strain-induced phase transformations of stainless steels

During tension or cold rolling of steel 301LN, the α' grains have been observed along thin bands that are partially composed of ε -martensite. Based on the previous section, it is believed that these bands were originally thin bands of ε -martensite, which transformed later on into α' -martensite. Therefore, the orientation of the α' -martensite is related to the orientation of the ε -martensite. Figure V.16 presents the orientations of the different phases present in one particular grain of steel 301LN cold-rolled to $\varepsilon = 0.16$. All pole figures are drawn in the austenite coordinates system of this particular grain. Figure V.16 (a) shows the band contrast map with the highlighted selected grain. Three systems of thin bands can be observed (ε_1 , ε_2 and ε_3). Figures V.16 (b), (c) and (d) present the IPF maps and the pole figures of the austenite, ε - and α' -martensite, respectively. Due to the low indexation, the orientation of only one ε variant (ε_1) appear on the pole figure. Four α' variants are formed in this grain.



(a)

*Mechanisms of Strain-induced Phase Transformations
in Austenitic Stainless Steels and Fe-Mn-Si-Al Steels*



*Figure V.16 : EBSD maps of
steel 301LN cold rolled
to $\varepsilon = 0.16$:
(a) band contrast map;
IPF maps (from Figure V.9)
and pole figures of :
(b) austenite ;
(c) ε -martensite ;
(d) α' -martensite.*

Figure V.17 presents the positions of the poles of ϵ -martensite (Figure V.17(a)) or α' -martensite (Figure V.17(b)) with the theoretical pole figure predicted by the KS relationships. One variant of ϵ -martensite is observed and its pole is superimposed with the $\{111\}$ pole of the austenite in the top right quadrant. Three α' variants (1, 2, 3) present a pole which is superimposed on the same spot. It indicates that these variants of α' -martensite respect the Burgers relationships with this ϵ variant. Based on the transformation mechanism presented in the previous section, it is believed that the corresponding grains of α' -martensite nucleated on the corresponding ϵ bands. One pole of the last α' variant (4) is found to superimpose on the $\{111\}$ pole of the austenite in the bottom right quadrant. Although the orientation of only one ϵ variant could be measured, two other systems of ϵ bands (*i.e.* two other ϵ variants) can be observed on the band contrast map (Figure V.16 (a)). It is likely that one of these two variants would present a pole that is superimposed on the austenitic $\{111\}$ pole in the bottom right quadrant. The grains corresponding to the α' variant number 4 would thus have nucleated on the bands corresponding to this unindexed ϵ variant. However, more experimental results would be required to confirm this hypothesis.

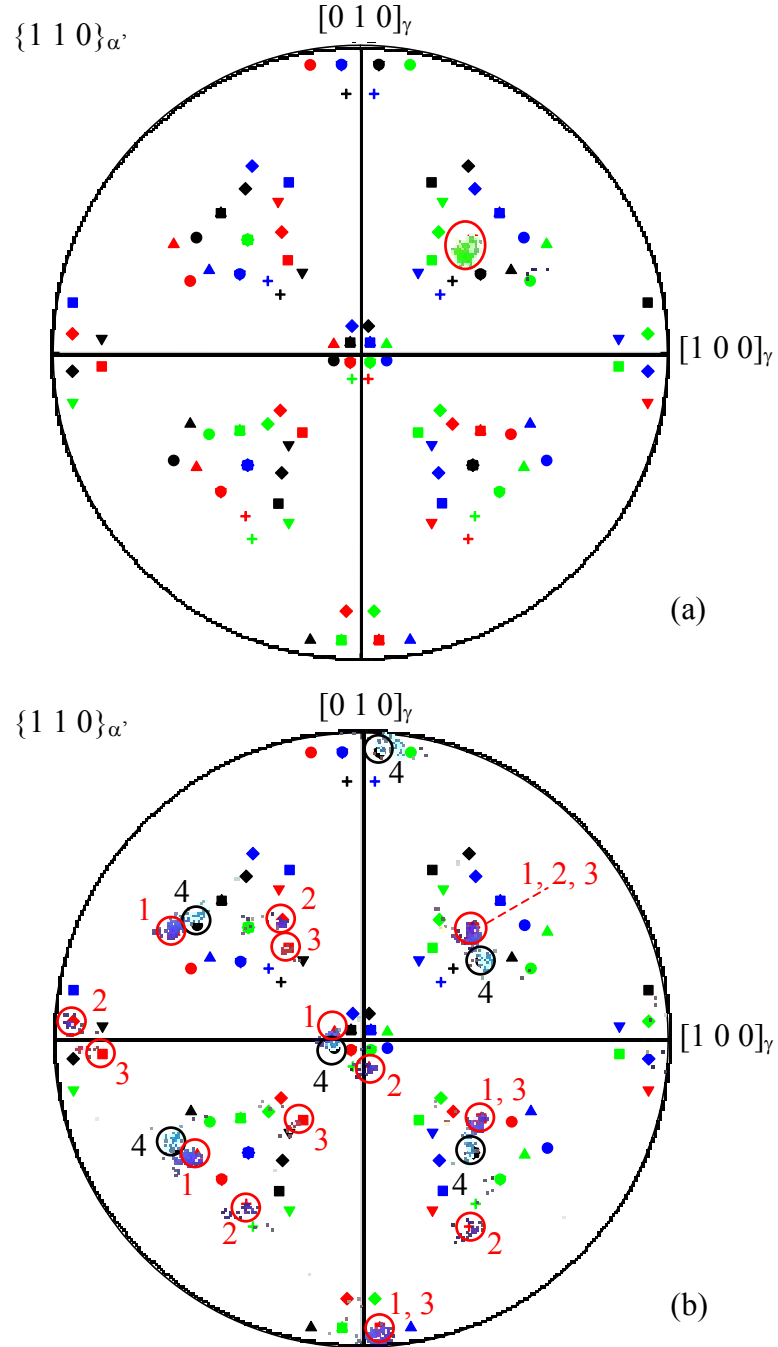


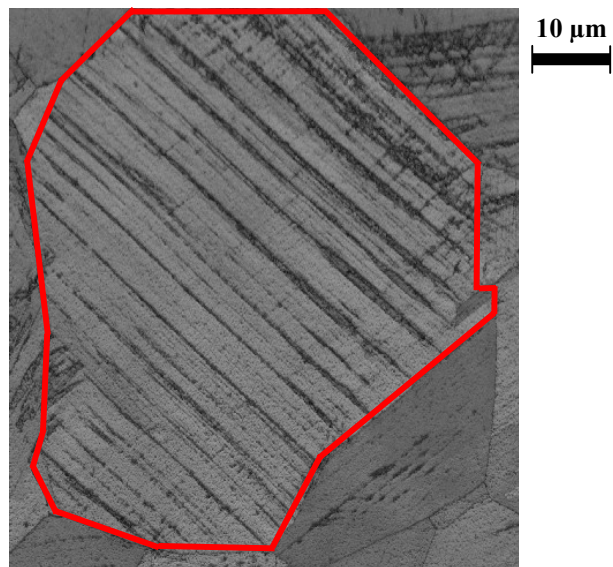
Figure V.17 : ε -martensite (a) and α' -martensite (b) pole figures of steel 301LN cold rolled to $\varepsilon = 0.16$, superimposed on the α' pole figure predicted by KS relationships.

The comparison of the α' pole figure with the KS prediction shows that some variants were formed preferentially. The selection of ε variants depends on the applied stress, as it has been explained in section V.3.1.1. As the α' -martensite nucleates on the ε -martensite, the formation of the ε bands dictates which α' variants are likely to be found. Within each variant of ε -martensite, six α' variants are predicted by the orientation relationships. However, it must be noted that only three α' variants are formed within the ε variant (Figure V.17 (b)). It can be assumed that the selection of these three α' variants among the six equally probable variants depends on the orientation of the transformation strain with respect to the externally applied stress. These phenomena have also been observed for 304 stainless steel samples deformed in tension at -60°C [Gey05].

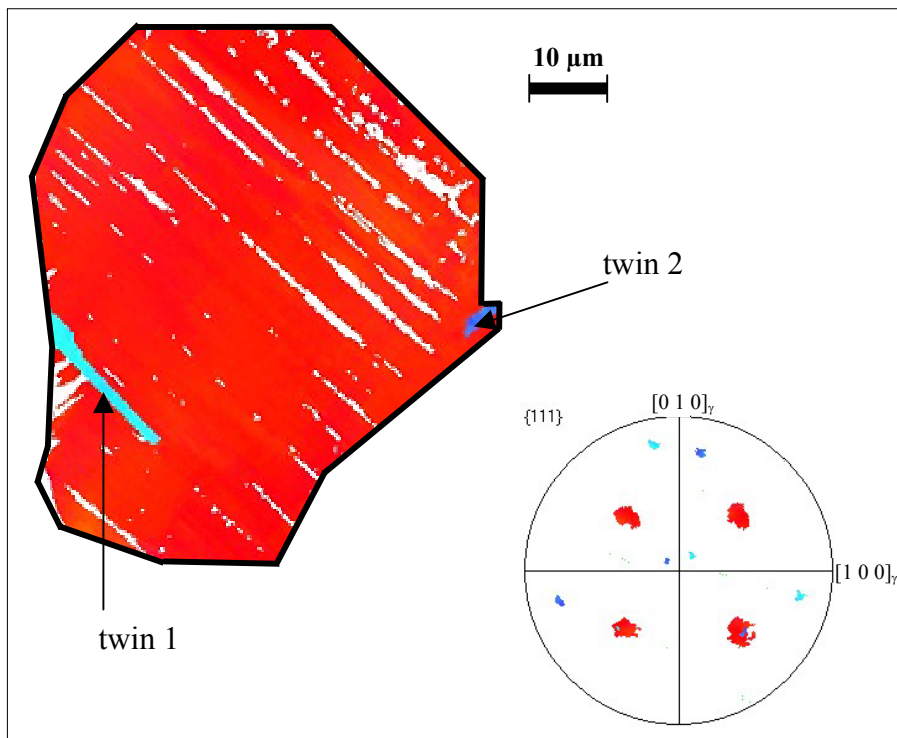
During rotary swaging of steel 301LN, it has been shown that the α' grains form along thin bands. Figure V.18 presents the orientations of the austenite and α' -martensite in one selected grain of steel 301LN strained by rotary swaging to $\varepsilon = 0.12$. All pole figures are drawn in the austenite coordinates system of this particular grain. Figure V.18 (a) presents the band contrast map with the selected grain in a red box. Figure V.18 (b) and (c) present the IPF map and the pole figure for the austenite and the α' -martensite, respectively. Two small annealing twins (twin 1, twin 2) can be observed in the austenite. It can be seen that the thin bands are parallel to one of these annealing twins, indicating that these bands probably appear along $\{1\ 1\ 1\}$ austenitic planes. Furthermore, an important misorientation spread can be observed within the α' -martensite.

The positions of the α' -martensite poles on the theoretical pole figure predicted by KS relationships is presented in Figure V.19. Five α' variants (1, 2, 3, 4, 5) exhibit one pole in common with the austenite $\{111\}$ pole in the bottom left quadrant, corresponding to one pole of the twin 1. These variants are found on the bands parallel to the twin 1 in Figure V.18 (b). Two other α' variants (6, 7) present one pole in common with the austenite $\{111\}$ pole of the bottom right quadrant, corresponding to the orientation of the austenite twin 2 in Figure V.18 (b). In order to keep the clearest figure while differentiating the variants, only two of the six poles of each variant have been labelled. The variant selection in rotary swaging is very difficult to determine as the true loading path is very complex and a large misorientation spread can be observed in the α' -martensite. The bands which can be observed along two $\{1\ 1\ 1\}$ austenitic planes play the same role as the ε bands in tension or rolling : all the α' variants formed by rotary swaging present one pole in common with one of the two system of thin bands. However, due to the complex loading path, the selection of two out of four $\{1\ 1\ 1\}$ austenitic planes can not be explained. However, almost all possible α' variants are formed within the most abundant band system, contrarily to cold-rolling. This could be explained by the multi-directional external stress. One possible consequence would be that, at some point, every variant within one band may find itself favourably orientated relatively to the externally applied stress.

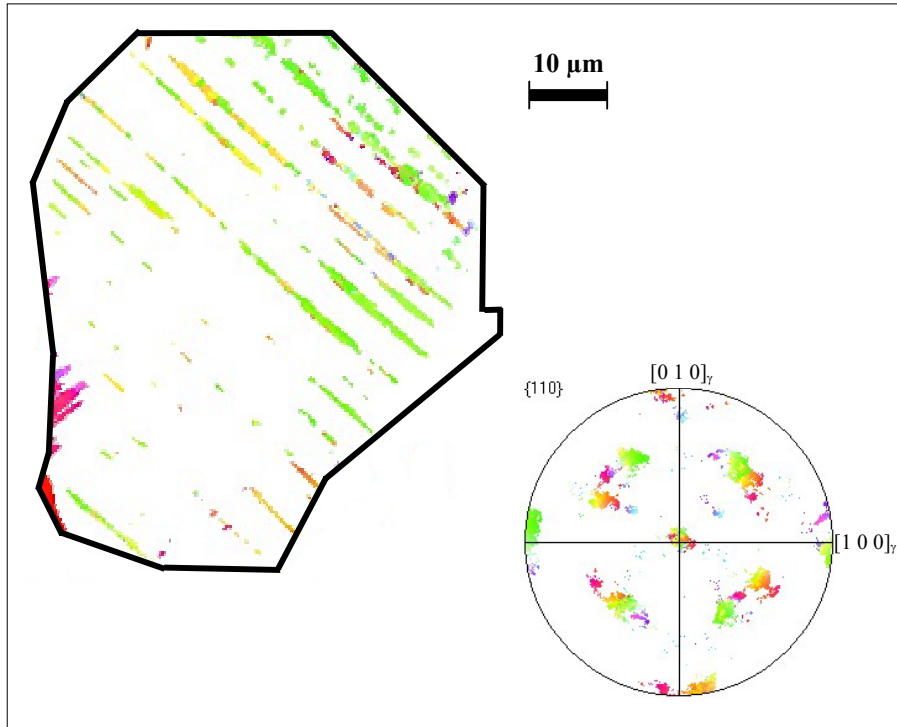
*Mechanisms of Strain-induced Phase Transformations
in Austenitic Stainless Steels and Fe-Mn-Si-Al Steels*



(a)



(b)



(c)

*Figure V.18 : EBSD maps of steel 301LN after
rotary swaging to $\varepsilon = 0.12$:
(a) band contrast map ;
IPF maps (from Figure V.10) and pole figures of
(b) austenite and (c) α' -martensite.*

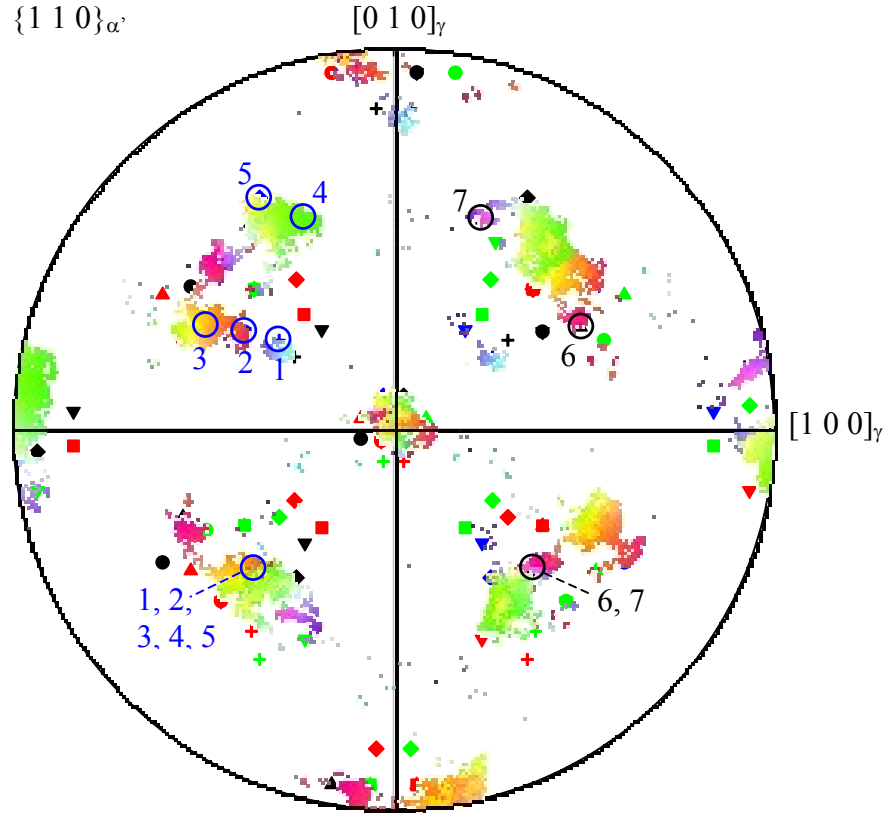


Figure V.19 : α' -martensite pole figure of steel 301LN strained by rotary swaging to $\varepsilon = 0.12$ superimposed on the α' pole figure predicted by the KS relationships.

V.4.2.2 Variant selection during strain-induced phase transformations of Fe-Mn-Si-Al steels

Figure V.20 shows the orientations of the different phases observed in a sample of steel Mn20 1000 strained by uniaxial tension to $\varepsilon = 0.16$. Figure V.20 (a) shows the band contrast map with the highlighted selected grain. Figures V.20 (b), (c) and (d) present the

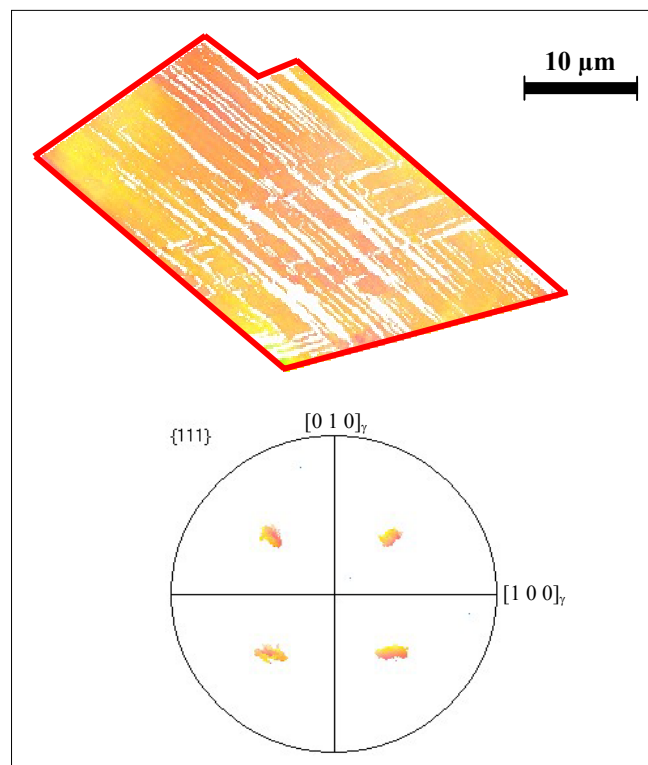
IPF maps and the pole figures of the selected area for austenite, ε - and α' -martensite, respectively. Two systems of ε bands can be observed on Figure V.20 (c) and the pole figure shows that each of the two ε variants has a pole in common with the austenitic mother phase. Several α' -martensite variants can be found (Figure V.20 (d)) and all have one pole in common with each ε variant. Moreover, some α' variants exhibit a twin relationship with each other. A misorientation spread is observed in all phases, which can be related to the stretch of the poles on the pole figures.

Figure V.21 compares the ε - and α' -martensite pole figures in the austenite reference system (of the selected area from steel Mn20 1000 of Figure V.20) with the α' theoretical pole figure predicted by the KS relationships. Each ε variant covers two poles of four KS variants (Figure V.21 (a)). These four KS variants correspond to the experimental α' variants (1, 2, 3 or 4) observed in steel Mn20 1000 (Figure V.21 (b)). As it has been mentioned, a twin relationship relates the KS variants 1 and 2, as well as 3 and 4. Therefore, the possible misorientations between these 4 variants are 10° , 50° or 60° (Table V.2). The orientation of the α' variants observed in steel Mn20 1000 are spread continuously over a couple of KS variants presenting a misorientation of 10° with each other, as it is shown by Figure V.21 (b).

*Mechanisms of Strain-induced Phase Transformations
in Austenitic Stainless Steels and Fe-Mn-Si-Al Steels*



(a)



(b)

*Mechanisms of Strain-induced Phase Transformations
in Austenitic Stainless Steels and Fe-Mn-Si-Al Steels*

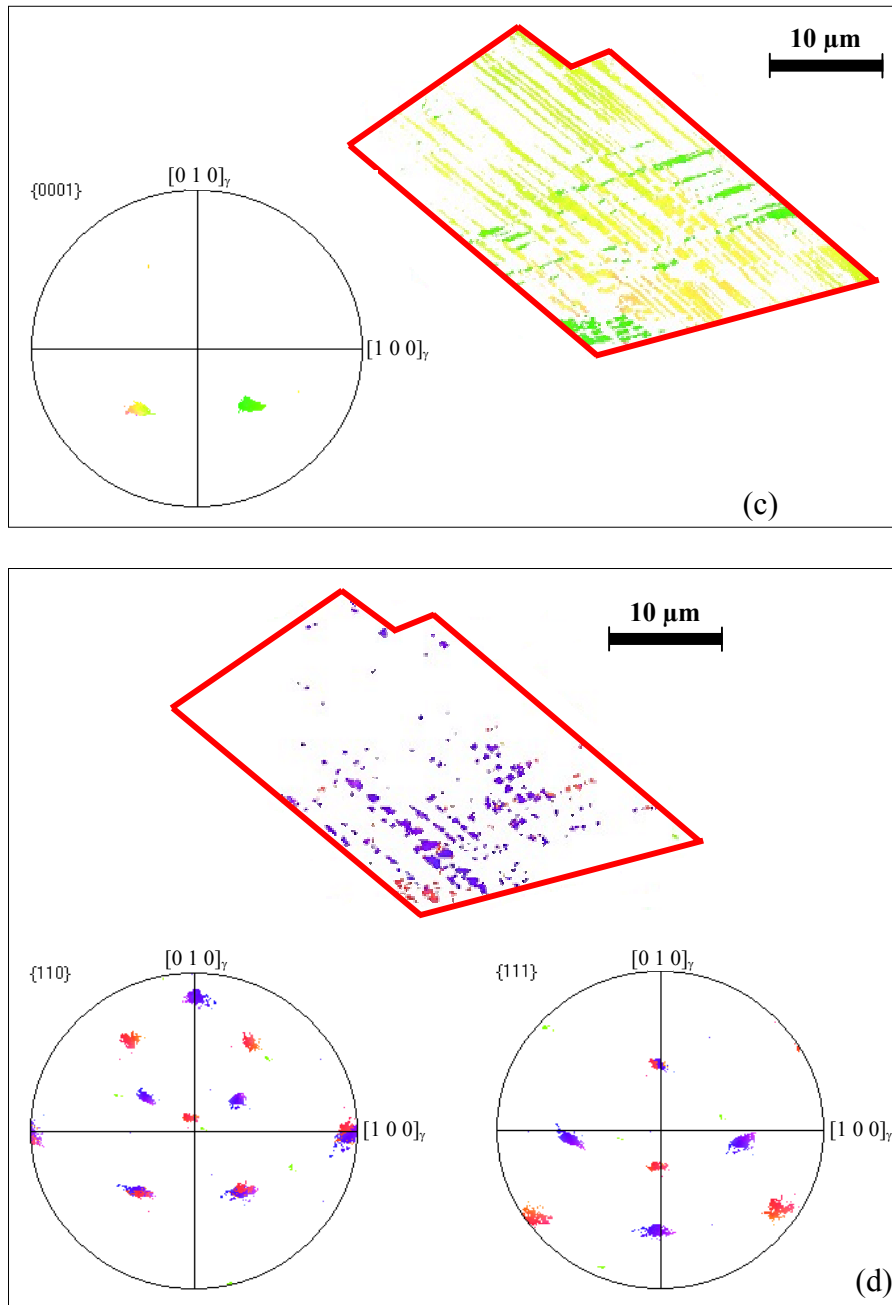


Figure V.20 : EBSD map of steel Mn20 1000 strained by tension to $\varepsilon = 0.16$: (a) band contrast ; IPF maps (from Figure V.3) and pole figures of (b) austenite; (c) α' -martensite and (d) ε -martensite.

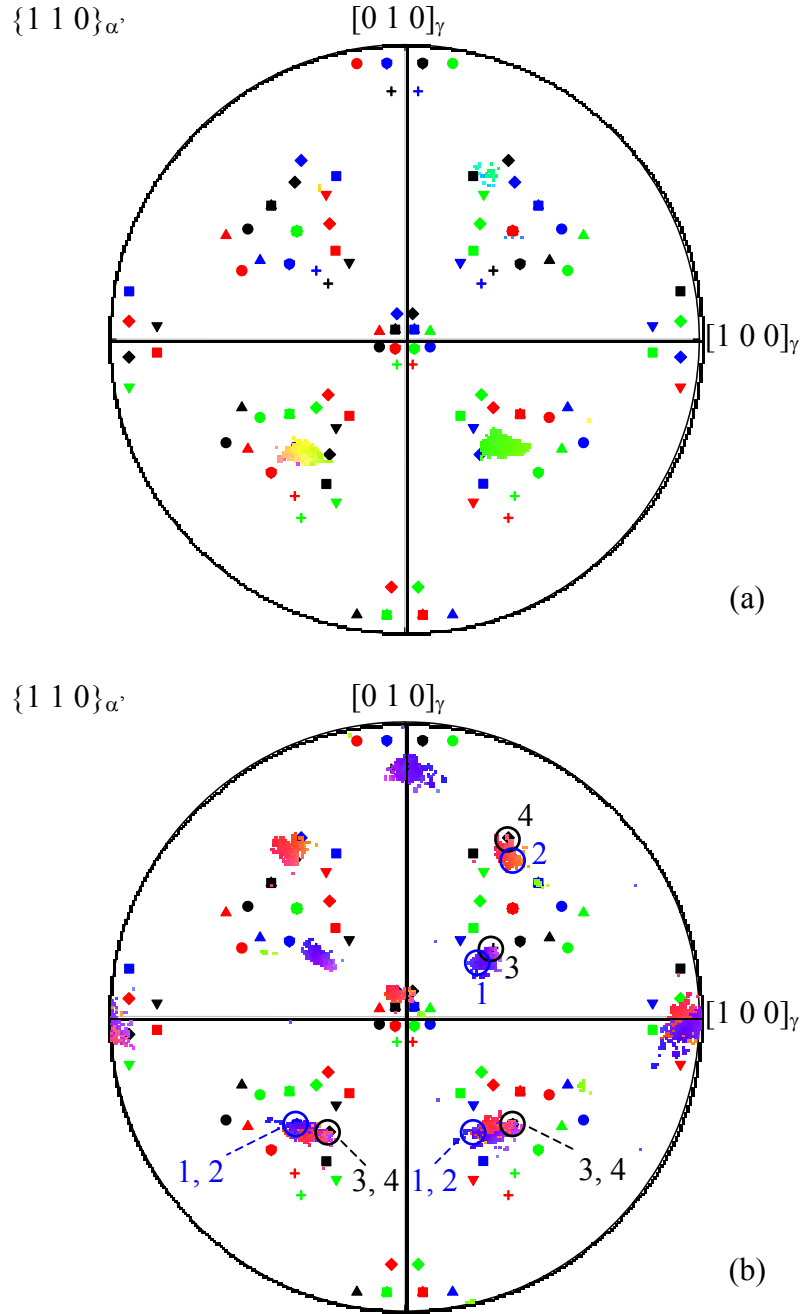


Figure V.21 : Superimposition of (a) the ε -martensite and (b) the α' -martensite pole figures of steel Mn20 1000 strained to $\varepsilon = 0.16$ on the α' pole figures predicted by the KS relationships.

*Mechanisms of Strain-induced Phase Transformations
in Austenitic Stainless Steels and Fe-Mn-Si-Al Steels*

	1	2	3	4
1	-	60°	10°	50°
2	60°	-	50°	10°
3	10°	50°	-	60°
4	50°	10°	60°	-

*Table V.2 : Angles between the 4 KS variants observed
in steel Mn20 1000 after tensile straining to $\varepsilon = 0.16$.*

The α' variant selection depends on the active ε systems and on the orientation relationships with two ε variants. As in stainless steels, the selection of ε -martensite variants depend on the orientation of the grain with respect to the externally applied stress. In steel Mn20, the α' -martensite grains nucleate nearly exclusively at the intersections of ε -martensite bands and grow strictly within the ε phase. Misorientation spreads are observed in all phases, bringing about a pole stretch in the pole figures. As a consequence, the poles of each ε variants are spread over the poles of two pairs of KS variants. In the sample, each α' variant has one pole in common with each of the ε variants. In other words, it means that besides the classic orientation relationship presented in Chapter I, each α' variant also respect this orientation relationship with the other ε variant in steel Mn20 1000. It is believed that these double crystallographic relationships are at the origin of the internal strain in all the phases. Moreover, as only two pairs of KS variants can fulfil these orientation conditions, the

crystallographic relationships completely dictate the α' variant selection for the $\varepsilon \rightarrow \alpha'$ phase transformation.

V.4.3 Emergence of secondary variants

Based on a hard spheres model, Yang and Wayman [Yang92a] have shown that the intersection of two bands of ε -martensite may bring about different atomic configurations, corresponding to HCP or FCC structures. It has been shown in Chapter I that the transformation of the austenite to a specific variant of ε -martensite can be described by the passage of a $1/6 \langle 11\bar{2} \rangle$ partial dislocation (creating an intrinsic stacking fault) every other $\{111\}$ planes. This is equivalent to the application of a homogeneous shear $1/12 \langle 11\bar{2} \rangle$ on $\{111\}$ planes (corresponding to half a twinning shear or $T/2$ shear), followed by a reorganization of atoms in every second basal plane. Yang and Wayman [Yang92a] showed that the formation of secondary variants is always associated with the intersection of two initial ε variants or shear systems, which corresponds to what is observed in the samples of steel Mn20. If two ε variants cross each other, their intersection corresponds to a volume of austenite that underwent a double shear.

Because of the orientation relationships between the austenite and the ε -martensite, this double shear may bring about the formation of an austenitic volume that would be undistorted but rotated in comparison with the original austenitic lattice. Indeed, it is known that during homogeneous shear of a lattice, one plane is undistorted : the basal shear plane. Moreover, a second plane remains undistorted by

the shear, although it is rotated to a new position : the conjugate shear plane. In case of $\gamma \rightarrow \epsilon$ transformation and due to the orientation relationships, the shear must be of the same angle for all variants and the shears bringing about two different variants must be applied on the same base plane or one on the conjugated plane of the other. Therefore, if a second homogeneous shear of the same amplitude is applied on a sheared lattice (due for example to the crossing with another ϵ variant), geometrically, there are two opposite shear directions for the second shear : one of them will normally cause an additional shear strain to the sheared lattice ; the other will produce a “negative” shear strain to the sheared lattice so that it is sheared back to its original lattice. It is of interest to note that the sheared lattice can be restored to its original structure not only by a reverse shear on the basal plane but also by a second shear on the conjugate plane. These two modes of restoring the distorted lattice produce two orientations which are related to each other by rotation through the same angle as between conjugate plane positions before and after shear. In other words, a lattice will undergo a rigid-body-rotation if two conjugate shears are properly combined, as it is shown by Figure V.22. In this Figure, the intersection volume of two ϵ variants may be regarded as austenite undergoing two T/2 shears on two conjugate shear systems (e.g. $(-1-11)$ $[112]$ and (111) $[-1-12]$ in Figure V.22). Starting with a particular austenite orientation (Figure V.22 (a)), two identical intermediates (Figure V.22 (b) and (c)) can be obtained as a result of two T/2 shears on $(-1-11)$ $[112]$ and (111) $[-1-12]$, respectively. For the second shear (on the second conjugate shear plane), two opposite directions are possible for each intermediate, *i.e.* $[11-2]$ or $[-1-12]$ on

(111) for the first intermediate of Figure V.22 (b) and [112] or [-1-1-2] on (-1-11) for the second intermediate of Figure V.22 (c). Each shear results in a rotation of 19.47° of the second conjugate shear plane. For each transition path indicated by the arrows, the original FCC lattice recovers after the double shear, although its orientation has changed. The resulting FCC variants (Figure V.22 (d), (e) and (f)) are related to the original variant by $+19.47^\circ$ (counter-clockwise), 90° and -19.47° (clockwise) rotations, respectively.

Several possibilities of intersection of ϵ bands are schematically illustrated on Figure V.23 (from [Yang92a]), corresponding the hard sphere models presented in Figure V.22. Figure V.23 (a) shows the original intersection morphology, before the application of the shears. After the double shears, it can be observed that the austenitic structure remains unchanged in the intersection region – as the position of the two conjugate planes remain unchanged – but can be considered as “rigid-body-rotated” of 90° (Figure V.23 (b)), -19.47° (Figure V.23 (c)) or 19.47° (Figure V.23 (d)). These correspond to the situations described in Figure V.22 (d), (b) and (c), respectively. Figure V.23 (e) shows the unfavourable configuration at the intersection, where all the planes are sheared into a position of type A.

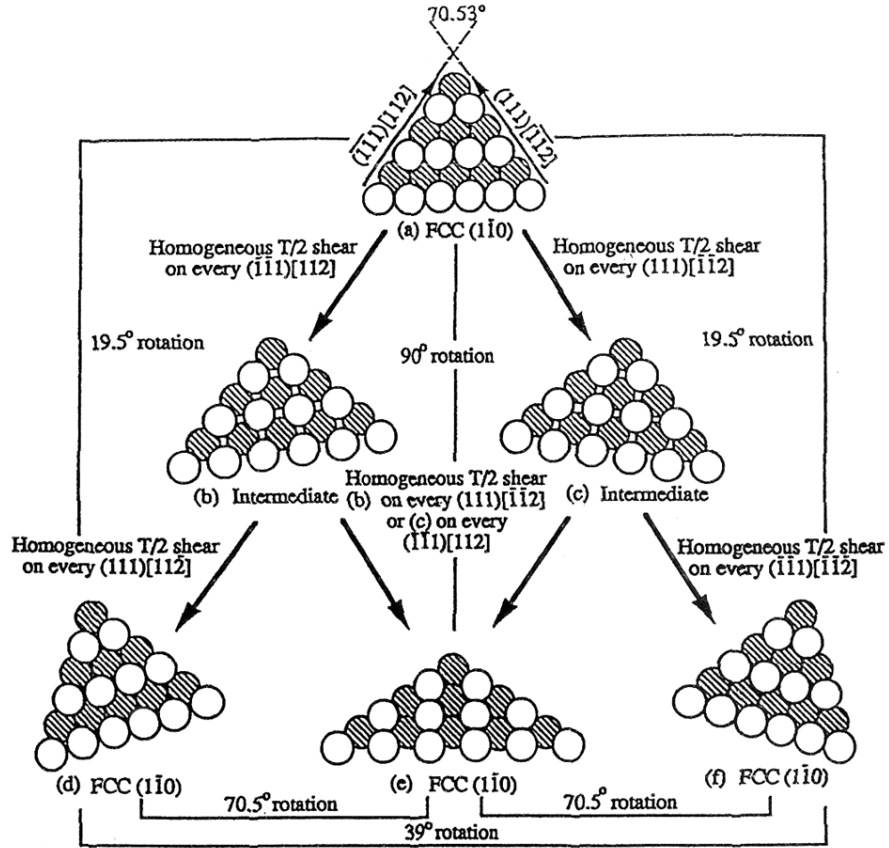
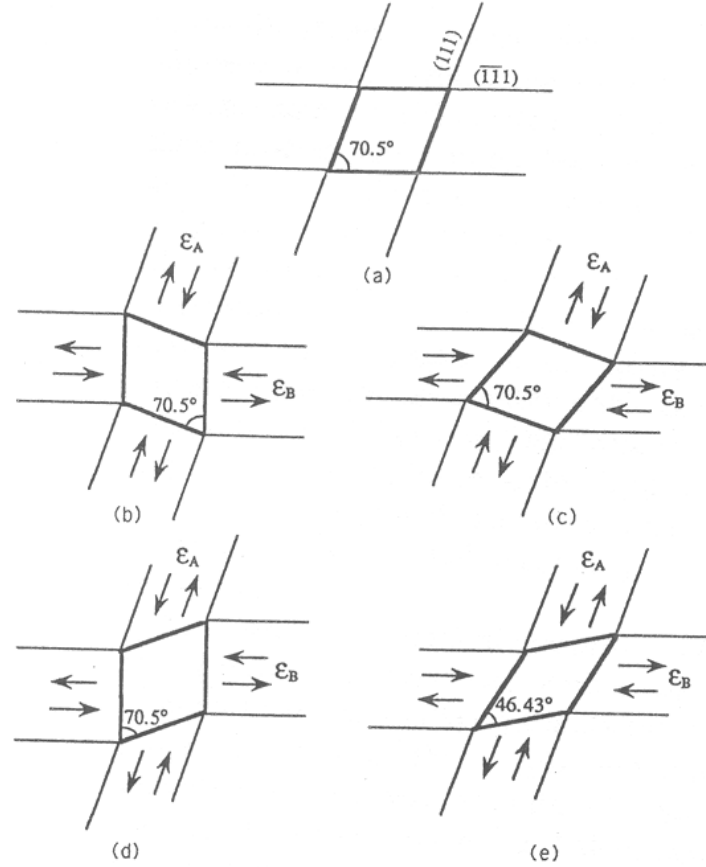


Figure V.22 : Hard sphere models illustrating rotation mechanisms for the orientation change of FCC variant by two intersecting shears ([1-10] projection from Thompson's tetrahedron) :
original morphology before the intersecting shears ;
(b) and (c) intermediate stages after a single shear ;
resulting F.C.C. structure at the doubly sheared intersections,
corresponding to a rigid-body rotation of the original austenite of (d)
19.47° ; (e) 90° or (f) -19.47° (from [Way92b]).

*Mechanisms of Strain-induced Phase Transformations
in Austenitic Stainless Steels and Fe-Mn-Si-Al Steels*



*Figure V.23 : Schematic illustration of intersections
of two ϵ variants (from [Way92a]) :*

*(a) original morphology before the intersecting shears ;
resulting F.C.C. structure at the doubly sheared intersections,
corresponding to a rigid-body rotation of the original austenite
of (b) 90° ; (c) -19.47° or (d) 19.47° .*

*The intersection shown in (e) corresponds to the unfavourable
stacking where all planes are in a position of type A.*

*The bold lines outlining the intersection region represent the new
positions of the two $\{111\}$ planes after the intersecting shears.*

In order to reduce the interfacial misfit between the different lattices, the transformation of the secondary γ variant into secondary ε variants is energetically favourable [Yang92a, Yang92b]. This transformation could take place after the formation of secondary γ variants, but also at some point during the intersection shears. All the possible secondary ε variants would then be related to the primary ε variants by one of six possible simple rotations : 19.47° , 31.59° , 38.94° , 51.06° , 70.53° and 90° [Yang92b]. In Figure V.12, the secondary ε variants are found at the intersection of two ε bands as well as next to the secondary austenite grains at these intersections. Therefore, it appears that the reduction of interfacial misfit was performed by two mechanisms : by the transformation of the secondary γ variant into a secondary ε variant, as proposed by Yang and Wayman, or by a shear of the surrounding ε bands, inducing a twinning process which brings about the secondary ε variant. Finally, a misorientation spread is observed in both austenite and ε -martensite so the poles of the primary variants and of the secondary variants actually touch each other, as it is shown on the pole figures of Figure V.13. This highlights the internal deformation of the grains and laths, probably caused by the orientation misfit at the interfaces between primary and secondary variants.

V.5 Conclusion

The crystallographic mechanisms of phase transformation in stainless steel 301LN as well as in steel Mn20 were investigated in

this Chapter. A general mechanism involving two steps can be observed in steel Mn20 :

1. The austenite transforms into ϵ -martensite, which forms as bands along $\{1\ 1\ 1\}$ austenitic planes.
2. The α' -martensite nucleates within bands of ϵ martensite, generally at the intersection of two bands. Later on, the α' grains grow strictly within the ϵ phase.

Based on the experimental results and on the literature [Lee01a, Lee01b, Spen04, Gey05], the same mechanism is believed to take place during phase transformations of stainless steel 301LN, although only a small fraction of ϵ -martensite could actually be observed. Indeed, most of it had already transformed into α' -martensite. However, in stainless steels, the α' grains grow within the ϵ bands but also directly in the austenite grains. Direct austenite to α' -martensite transformation can thus also be observed.

In steel Mn20, the orientation of ϵ bands correspond to the most favourably oriented planes for gliding of dislocations relatively to the externally applied stress, as it was shown by OIM analysis. This tend to confirm the mechanisms of formation by piling up of stacking faults created between the partial dislocations on $\{1\ 1\ 1\}$ planes.

Besides the general mechanism, two particular cases have been observed : the influence of deformation temperature during rotary swaging in stainless steels and the formation of secondary austenite and secondary ϵ -martensite in Fe-Mn-Si-Al steels :

1. after deformation by rotary swaging, no ϵ -martensite could be observed in stainless steels due to the rise of sample temperature during deformation. However, thin bands are still observed along $\{1\ 1\ 1\}$ austenite planes. The α' -martensite grains nucleate on these bands which could be ϵ -martensite too thin to be indexed, shear bands or thin mechanical twins.
2. in some more rare occasions, the intersection of two ϵ bands in Fe-Mn-Si-Al steels brought about secondary variants of austenite or of ϵ -martensite. The transformation of austenite into ϵ -martensite is equivalent to a shear of the austenite lattice, followed by reorganisation of the atoms every other basal plane. The double shear observed at junctions of two ϵ bands may bring about the formation of small grains of secondary austenite. The secondary austenite corresponds to a rigid-body rotation of the austenite mother lattice. In order to reduce the interface misfit, the grain of secondary austenite or a part of the band of ϵ -martensite at the boundary transforms by shear into secondary ϵ -martensite, which exhibits a twin relationship with the band of primary ϵ -martensite. Moreover, a local strain induces a misorientation spread, which reduces the interface misfit between primary and secondary variants in both phases.

The comparison between the results and the crystallographic orientation relationships show that some variants of ϵ - and α' -martensite are formed preferentially. The variant selection phenomenon takes place in two steps :

- 1) formation of ϵ variants as a function of their relative orientations to the externally applied stress. This phenomenon is common to stainless steels and Fe-Mn-Si-Al steels.
- 2) selection of α' variants among the six possible variants in each band of ϵ -martensite :
 - in stainless steels, the α' variant selection depends on the crystallography of the transformations and on the relative orientations of the transformation strain to the local stress field. However, no variant selection is observed in rotary swaging as the loading direction changes continuously.
 - in Fe-Mn-Si-Al steels, the α' variant selection is dictated by crystallographic considerations only. Indeed, large misorientation spreads are observed within the ϵ bands and the α' grains. These spreads indicate a local deformation of the lattice, required to respect the orientation relationships between the intersecting laths of ϵ -martensite which are crossing and the nucleating α' grains. As a consequence, the orientation of each α' variant found in Fe-Mn-Si-Al steels is spread over the orientation of two variants predicted by the Kurdjumov-Sachs (KS) relationships with a misorientation of 10° . With respect to the misorientation spread within the ϵ -martensite, only two couples of KS variants respect the orientation relationships with both ϵ variants. These two couples correspond to the two α' variants observed in Fe-Mn-Si-Al steels.

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*Mechanisms of Strain-induced Phase Transformations
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