



## Full length article

## Misorientation distribution between martensite and austenite in Fe-31 wt%Ni-0.01 wt%C

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## ABSTRACT

We characterized the morphology, substructure and crystallography of lenticular martensite in a Fe-Ni-C alloy by means of electron backscatter diffraction and scanning electron microscopy. Electron backscatter diffraction maps were used to determine the orientation relationship between austenite and martensite across large regions of the microstructure. We employed orientation distribution functions as a statistical representation method for the observed orientation relationships. High-resolution point-to-point scans were used to normalize the effects of the orientation changes in the austenite caused by the plastic deformation during the formation of lenticular martensite. The analysis revealed that most of the transformation in this material follows an orientation relationship close to the one proposed by Greninger and Troiano.

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## 1. Introduction

The martensitic transformation is a solid-state non-diffusional first-order reaction in which the parent phase undergoes both a structural and a shape change. The strain energy associated with the shape change plays an important role for the morphology and crystallography of the transformed phase [1–3]. Examples of martensitic transformations and their applications can be found in several areas, including the metallurgy of metallic alloys and biological systems (i.e. certain motor proteins in viruses and bacteria) [4]. The greatest industrial relevance of the martensitic transformation lies in its role as the most important hardening mechanism in steels and titanium alloys.

Martensite in steels shows diverse morphologies depending on the chemical composition of the specific alloy and consequently on its martensite transformation start temperature,  $M_s$ . At higher  $M_s$  temperatures, martensite tends to form as laths, whereas at lower  $M_s$  temperatures martensite tends to form as plates that can be thin or lenticular.

Each chemical composition and heat treatment path results in specific substructures and crystallography [5,6]. Lenticular martensite has a complex substructure formed at intermediate  $M_s$  temperatures [7–11]. It is characterized by three distinct regions. The first is the central midrib region constituted by  $\{112\} \langle \bar{1}\bar{1}1 \rangle$  twins. In the second region, some of the twins extend beyond the midrib and coexist with screw dislocations. The third region is untwinned and comprises dislocation tangles.

Regarding the orientation relationship (OR) between austenite and martensite, several crystallographic models have been proposed [12–17]. The main ORs proposed and/or observed are summarized in Table 1. According to some authors [18–21], in lenticular martensite, the OR between the midrib region and the austenite is closer to Greninger-Troiano (GT) whereas the OR between the untwinned regions of the martensite (in the vicinity of the  $\alpha'/\gamma$  interfaces) and the austenite is closer to Kurdjumov-Sachs (KS). These authors also report that the habit plane changes from  $\{3\ 10\ 15\}$  to  $\{111\}$  or  $\{225\}$  from the midrib to the  $\alpha'/\gamma$  interface. As reported by Cohen and Wayman [1], the growth of the martensite midrib is faster and precedes the growth of the lenticular region. Shibata et al. [19] showed that the midrib region can be regarded as a thin plate martensite. The heat released by the exothermic martensitic transformation locally raises the temperature around

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**Table 1**

Most usual orientation relationships (ORs) between face centered and body centered crystals.

Name	OR	(uvw) $\omega_{\min}$	Euler angles		
			$\varphi_1$	$\Phi$	$\varphi_2$
Bain [12]	$\{100\}_{\gamma}/\{100\}_{\alpha}$ $\langle 100 \rangle_{\gamma}/\langle 110 \rangle_{\alpha}$	$\langle 100 \rangle$ 45°	0°	45°	0°
KS [13]	$\{111\}_{\gamma}/\{110\}_{\alpha}$ $\langle 110 \rangle_{\gamma}/\langle 111 \rangle_{\alpha}$	$\langle 0.97 \ 0.18 \ 0.18 \rangle$ 42.85°	5.77°	48.19°	5.77°
NW [14,15]	$\{111\}_{\gamma}/\{110\}_{\alpha}$ $\langle 112 \rangle_{\gamma}/\langle 110 \rangle_{\alpha}$	$\langle 0.98 \ 0.08 \ 0.20 \rangle$ 45.98°	0°	45°	9.73°
GT [16]	$\{111\}_{\gamma}/\{110\}_{\alpha}$ $\langle 123 \rangle_{\gamma}/\langle 133 \rangle_{\alpha}$	$\langle 0.97 \ 0.19 \ 0.13 \rangle$ 44.23°	2.7°	46.6°	7.5°
Pitsch [17]	$\{100\}_{\gamma}/\{110\}_{\alpha}$ $\langle 110 \rangle_{\gamma}/\langle 111 \rangle_{\alpha}$	$\langle 0.08 \ 0.20 \ 0.98 \rangle$ 45.98°	9.73°	45°	0°

the midrib, allowing the transformation to proceed through slip [19].

Previous studies highlighted the advantages of EBSD over TEM diffraction methods in the study of ORs between martensite and austenite [20–22]. In this context the main limitation of TEM is its small area of observation. As a consequence, TEM observations cannot be used to collect larger amounts of crystallographic data that would be sufficient for conducting a statistically robust analysis. In contrast, other techniques such as synchrotron or neutron diffraction allow the investigation of larger volume fractions, but these techniques require single crystals or very coarse grained samples to arrive at meaningful insights on the underlying ORs and at spatial correlations. These techniques have for instance been employed to study ORs in iron-based meteorites owing to their coarse grain size [23].

Most EBSD studies that were conducted on the ORs of lath martensite [20,21] have been performed focusing on individual laths. Nonetheless, the EBSD technique may also be used to scan large microstructural areas producing an amount of results that permits conducting both, full statistical and spatially correlated analysis [22].

There are a number of commonly used ways to represent and analyze ORs, usually based on the martensite-martensite misorientation. These are for instance the pole figures (PF) of the martensite variants belonging to the same parent austenitic grain; the misorientation histograms between the variants showing the distribution of the rotation angles between the variants and also the rotation axes in inverse pole figures (IPF); the representation of the angle between the close-packed directions (CPD) and close-packed planes (CPP) of the fcc and bcc phases in a (CPD, CPP) graph [24]; and vectors in the Rodrigues-Frank space [25,26]. All these methods are based on the comparison of the martensite-martensite misorientation relationship and should be distinguished from other methods based on the calculation and reconstruction of the austenitic orientation [24,27,28].

Traditionally, pole figures are the most used representation type for analyzing and representing orientation relationships but the angular differences among the different postulated ORs are so small that it is very difficult to distinguish between them using discrete PFs. A more robust analysis of the ORs can be made by representing them in terms of Euler angles in a three-dimensional orientation space, as proposed by Nolze [29,30]. According to some authors [29–32], representations in Euler space can be more accurate than two-dimensional projections such as pole and inverse pole figures for the analysis of the ORs because even minor changes in the misorientation angle or in any of the three symmetry axes of the coordinate system can be readily and independently identified. Also, the use of Euler angles is more intuitive so that the Orientation Distribution Function (ODF) is an adequate means of representing

crystallographic textures and misorientation distributions [31,32].

In this paper, the orientation relationship between lenticular martensite and austenite is investigated. For this purpose, we propose to employ Euler angles using Bunge's notation [31,32] to represent martensite–austenite misorientation distributions. For obtaining an adequate data basis we used the EBSD technique to map large areas of martensite-austenite microstructures in polycrystalline samples. These mappings provide large data sets that enable us to perform a statistically robust analysis of the ORs developed between martensite and austenite crystals.

## 2. Experimental procedure

### 2.1. Material

Fe-31 wt%Ni-0.01 wt%C (% refers to weight % here and throughout) alloy samples were produced by means of vacuum induction melting, hot rolling and vacuum annealing for 18 h at 1373 K [33]. The material in this condition is fully austenitic with a grain size obtained as mean intercept length of 16.8  $\mu\text{m}$  [34]. The bulk chemical composition of this alloy is shown in Table 2 [35]. This alloy has several advantages for crystallographic studies related to the martensitic transformation: (a) the transformation occurs at temperatures below room temperature, allowing characterization of the austenitic microstructure at room temperature before the reaction. (b) depending on the transformation temperature a high fraction of austenite is retained, which allows the direct measurement of the orientation relationships between  $\alpha'$  and  $\gamma$ ; (c) the very low carbon content is present in this alloy to inhibit the undesired formation of strain-induced martensite during metallographic preparation.

A “fully transformed” sample was obtained by cold rolling and vacuum annealing at 1573 K for 3600 s (1 h), resulting in austenite with a coarse a grain size (mean intercept length) equal to 106.4  $\mu\text{m}$ . Subsequent cooling down to liquid nitrogen temperature (77 K) yields 90 vol.% of martensite transformation [34]. A partially transformed sample was obtained by cold rolling and vacuum annealing the alloy at 1073 K for 3600 s obtaining an austenitic grain size (mean intercept length) equal to 19.6  $\mu\text{m}$ . Cooling the samples down to 212 K yields about 8 vol % of martensite [34].

**Table 2**

Chemical composition of the studied alloy (in %wt.) [35].

C	N	Ni	Ti	Zr	Si
0.007	<0.00009	31.5	<0.005	<0.01	<0.005
Cu	Co	Cr	V	Fe	
0.01	0.008	<0.003	<0.005	Bal.	

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