



PROCEEDINGS OF THE INTERNATIONAL CONFERENCE ON MARTENSITIC TRANSFORMATIONS: **CHICAGO**

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The Minerals, Metals & Materials Series

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Preface

The International Conference on Martensitic Transformations (ICOMAT) was held on July 9–14, 2017, at the Hyatt Regency in Chicago, Illinois, USA. The event was hosted by the Chicago-based CHiMaD Center for Hierarchical Materials Design in downtown Chicago.

Located on the shores of Lake Michigan in the heart of the Great Lakes Region of North America, the city of Chicago is truly an epitome of the “melting pot” reputation of the United States, evidenced by a diverse array of cultural, economic, and social experiences that draw tourism, science and technology, arts, and music to its streets year-round. The “Chicago School” represents a unique tradition of innovation across many fields, notably including materials, as the birthplace of Materials Science in the 1950s and Materials Design in the 1990s.

ICOMAT 2017 was organized around the central theme of “Martensite by Design.” Symposia included design for microstructures, properties, advanced manufacturing, and performance. Topics included:

- Theory & Methods for Martensite Design
- Interactions of Phase Transformations and Plasticity
- Quenching and Partitioning of Martensite and Other Advancements in Steels
- Novel Shape Memory Alloys
- Novel Functional Behaviors: Beyond Shape Memory Effect & Superelasticity
- Martensitic Transformations in Non-Metallic Materials
- Size Effects in Martensitic Transformations
- Advanced Characterization of Martensite—3D & High Resolution
- Quasimartensitic Modulations
- Advanced Processing Techniques: Additive, Porous, and Others
- Engineering Applications and Devices
- MSMnet: Magnetomechanics of Magnetic Shape Memory Alloys

The next ICOMAT conference will be held in 2020 in Jeju Island, South Korea.

Aaron P. Stebner
Gregory B. Olson

Correlation Between Deformation Texture and Martensitic Transformation in TWIP/TRIP Steels on Multiscale

Marton Benke, Erzsebet Nagy, Mate Sepsi, Peter Pekker, and Valeria Mertinger

Abstract

In the present manuscript, the texture variation of γ austenite, ε martensite and α' martensite phases are investigated in FeMn (Cr) steels exhibiting both TWIP and TRIP behaviour during uniaxial tensile tests. Samples of three steels with varying Cr content were subjected to tensile tests till fracture on different temperatures ranging from room temperature, at which ε martensite and γ austenite are stable to 453 K, where only γ austenite was present prior to tensile stressing. The developed texture of ε martensite, α' martensite and γ austenite was examined on multiscale, by TEM orientation mapping, Electron Backscattered Diffraction (EBSD) orientation mapping and X-ray-diffraction pole figure measurements. Correlations between the developed textures and the formation of the martensitic phases are discussed.

Keywords

TRIP/TWIP steels • Texture • Thermomechanical treatment

Introduction

TWIP/TRIP steels are favoured because of their combined high strength and elongation. In case of TWIP steels, the unique mechanical behaviour is due to Twinning Induced Plasticity (TWIP) which is based on the $\gamma \rightarrow \varepsilon$ martensitic transformation, while Transformation Induced Plasticity is characteristic to TRIP steels which is based on the $\gamma \rightarrow \alpha'$

α' and $\gamma \rightarrow \alpha'$ martensitic transformations [1–4]. The unique mechanical behaviour of TWIP/TRIP steels can be utilised in energy absorbing components and/or during the manufacturing process of such steels.

Principles of the transformations occurring in TWIP and TRIP steels such as crystallographic correlation during fcc-hcp transformations has been described for long [1–6]. However, because of the components' mechanical behaviour during deformation must be well known and controlled for proper process design, detailed examinations on the behaviour of TWIP/TRIP steel with specific compositions are still being investigated. For instance, Kwon et al. have shown that reverse $\varepsilon \rightarrow \gamma$ transformation can occur during deformation in high Mn-content steels [6]. Besides the deformation induced transformations, crystallographic texture has also effect on the components' formability. Barbier et al. examined the texture evolution during room temperature tensile tests on macroscale and microscale by means of X-ray diffraction (XRD) and electron backscattered diffraction (EBSD). They showed that a pronounced $\langle 111 \rangle$ fiber texture formed during tensile tests [7]. Co-authors of the present paper investigated the macrotexture evolution of TRIP steels at sub-zero temperature levels using XRD [8–10].

However, the description of texture formed at elevated temperatures is also useful for practical applications if the thermomechanical process of TWIP/TRIP steels occurs at elevated temperatures.

The aim of the present paper is to characterize the orientation relationships of γ austenite, ε martensite and α' martensite within the same former austenite grain in samples subjected to tensile tests at different temperatures both above and below the martensite start (M_s) temperature of the $\gamma \rightarrow \varepsilon$ transformation.

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Experimental

TWIP/TRIP steels with Cr content of 0.07, 2.26 and 6.12 were produced at TU Bergakademie Freiberg. The compositions of the examined steels are given in Table 1. Tensile test specimens with diameter of 5 mm and differential scanning calorimeter (DSC) samples were machined from hot rolled rods. The specimens were austenized at 1273 K in argon atmosphere for 30 min and subsequently quenched in room temperature water. XRD phase analysis confirmed that the microstructure of the as-quenched (AQ) samples consisted of thermally induced ϵ martensite and γ austenite of all three steel types (Table 2). The $\epsilon \leftrightarrow \gamma$ transformation temperatures were obtained using a Netzsch 204 heat flux DSC. The $\epsilon \leftrightarrow \gamma$ transformation temperatures are summarized in Table 1. Since the formation of α' martensite could be induced only by mechanical stress in the examined alloys, only the $\epsilon \leftrightarrow \gamma$ transformation temperatures were measured. Accordingly, martensite start temperature of the $\epsilon \leftrightarrow \gamma$ transformation is referred to as ' M_s '. Tensile tests were performed at different test temperatures with an Instron 5982 universal mechanical tester machine equipped with a climate chamber. The test temperature range was set to reach A_f temperatures and go below M_f temperatures. Prior to tensile tests, the specimens were heated up to 573 K and held for 30 min. After that, the specimens were cooled down to the test temperature being: 473, 453, 433, 413, 398, 383 and 313 K and loaded until rupture. The detailed anisotropy investigation of tested samples at 453, 398 K are introduced in this paper.

After tensile tests, samples for XRD and transmission electron microscope (TEM) examinations were prepared from the cross-gauge section of the tensile specimens. Specimens stressed above M_s consisted of γ austenite and strain induced ϵ martensite (ϵ_{STR}) on the test temperature. Specimens tensile tested below M_s contained thermally induced ϵ martensite (ϵ_{TH}) in addition to γ and ϵ_{STR} . α' martensite also formed in samples stressed at 413 K or lower temperatures. The volume fraction of phases was determined by full intensity fitting X ray diffraction method. Orientation relationships between γ austenite and ϵ martensite, adjacent ϵ plates and ϵ martensite and α' martensite crystals in nano scale were characterised by TEM orientation mapping using a FEI Tecnai G² TEM and ASTAR system. The orientation relationships were described through pole figure generations. For every orientation relation, the examined crystal pairs were located within a common former austenite grain. For each examined sample, examinations were carried out on 5 different fields of view. For the colour version of the TEM map images the Reader is asked to see the online version. The colours correspond to different orientations of crystals, thus, plates of the same phase with different orientations can be distinguished. The orientations related to different colours are not given since pole figures were used for the orientation investigations. The microscale orientation mapping was described by EBSD using Zeiss EVO MA 10 scanning electron microscopy equipped with EBSD system. The macroscale orientation was characterised by Bruker D8 Advance X ray diffractometer and Stresstech G3R centreless diffractometer using inverse pole figure and pole figure descriptions respectively.

Table 1 Composition and transformation ($\epsilon \leftrightarrow \gamma$) temperatures of the examined steels

Steel type	Composition						Transformation temperatures			
	C	Mn	Cr	Si	S	P	M_s	M_f	A_s	A_f
	wt%						K			
Steel 2	0.028	18	0.07	0.03	0.025	0.0081	411	319	455	499
Steel 1	0.026	17.7	2.26	0.1	0.029	0.0051	404	319	453	494
Steel 3	0.08	17.7	6.12	0.06	0.025	<0.003	378	310	444	481

Table 2 Volume fraction of phases, %

Steel type	Test temperature								
	As quenched			398 K			453 K		
	γ	α	ϵ	γ	α	ϵ	γ	α	ϵ
	%			%			%		
Steel 2	8	0	92	13	31	56	45	0	55
Steel 1	14	0	86	6	68	26	51	0	49
Steel 3	14	0	85	36	14	50	47	0	53

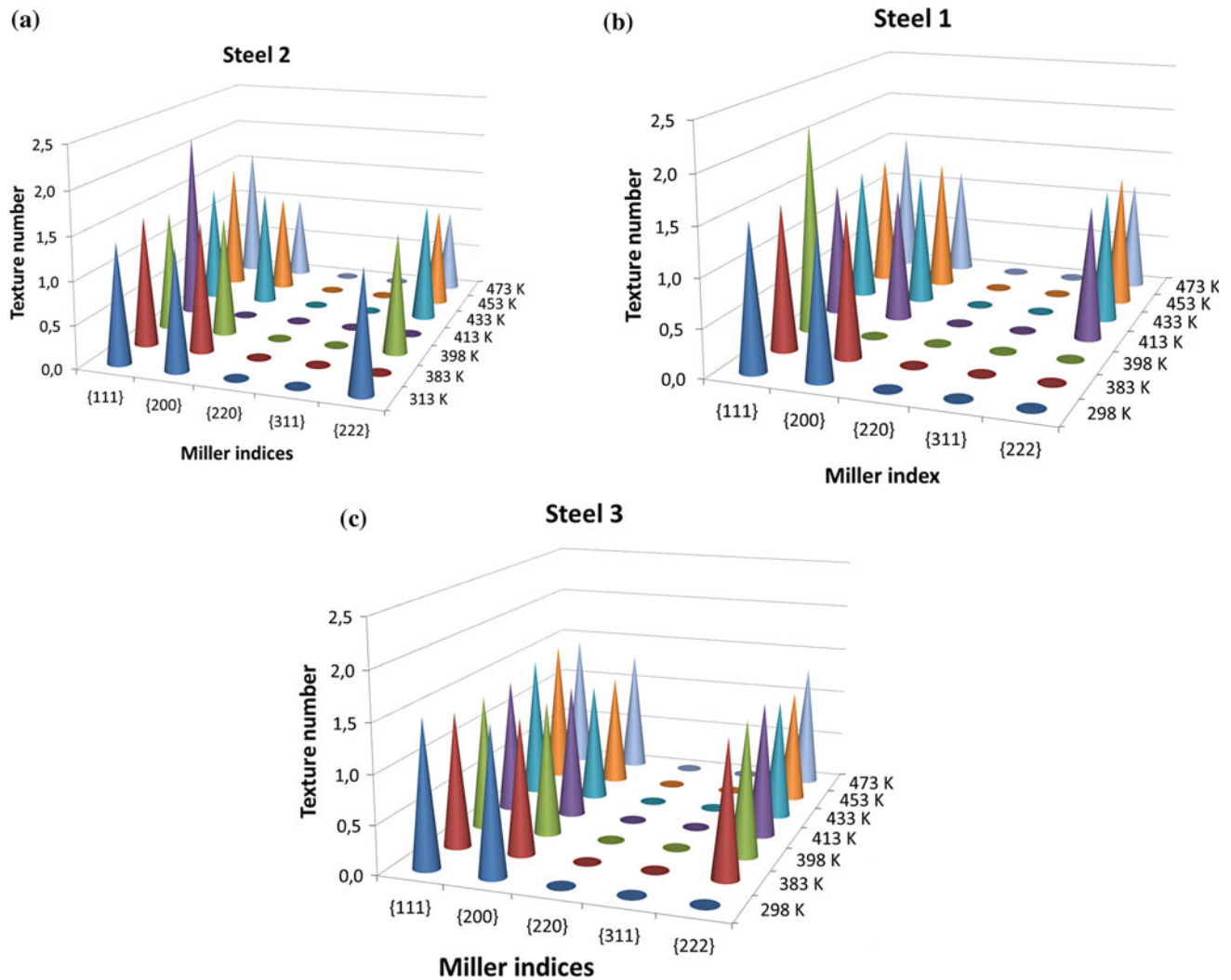


Fig. 1 Texture numbers of γ phase obtained from cross section XRD spectra of **a** steel 2, **b** steel 1 and **c** steel 3 tensile tested at different temperatures [11]

Results and Discussion

The Table 2 shows the calculated volume fraction for the fractured samples at 398 and 453 K. Austenite and ε martensite were determined at the higher temperature while the three phases mixture (austenite, ε and α' martensites) were found at the lower temperature. Formation of the strain induced martensite is evident at the 453 K in case of all steel types. In case of Steel 2 and Steel 1 the M_s temperature is higher than the lower test temperature so a mixture of thermally and stress induced ε martensite can be expected after fracture at 398 K. In case of Steel 3 this test

temperature is slightly above to the M_s temperature (378 K) so the thermally induced ε martensite is unexpected. The loaded at lower temperature resulted strain induced α' martensites in case of each steel.

The orientation relationships are performed in the rank of scale as it were determined.

Figure 1 and 2 show the texture numbers of γ and ε phases, respectively, calculated from the relative intensity ratios of Bragg reflections on cross section XRD spectra of the steel samples after tensile tests at different temperatures.

As seen in Fig. 1, the γ phase remained closely isotropic after all performed tensile tests regardless of test temperature or steel composition. However, according to Fig. 2,

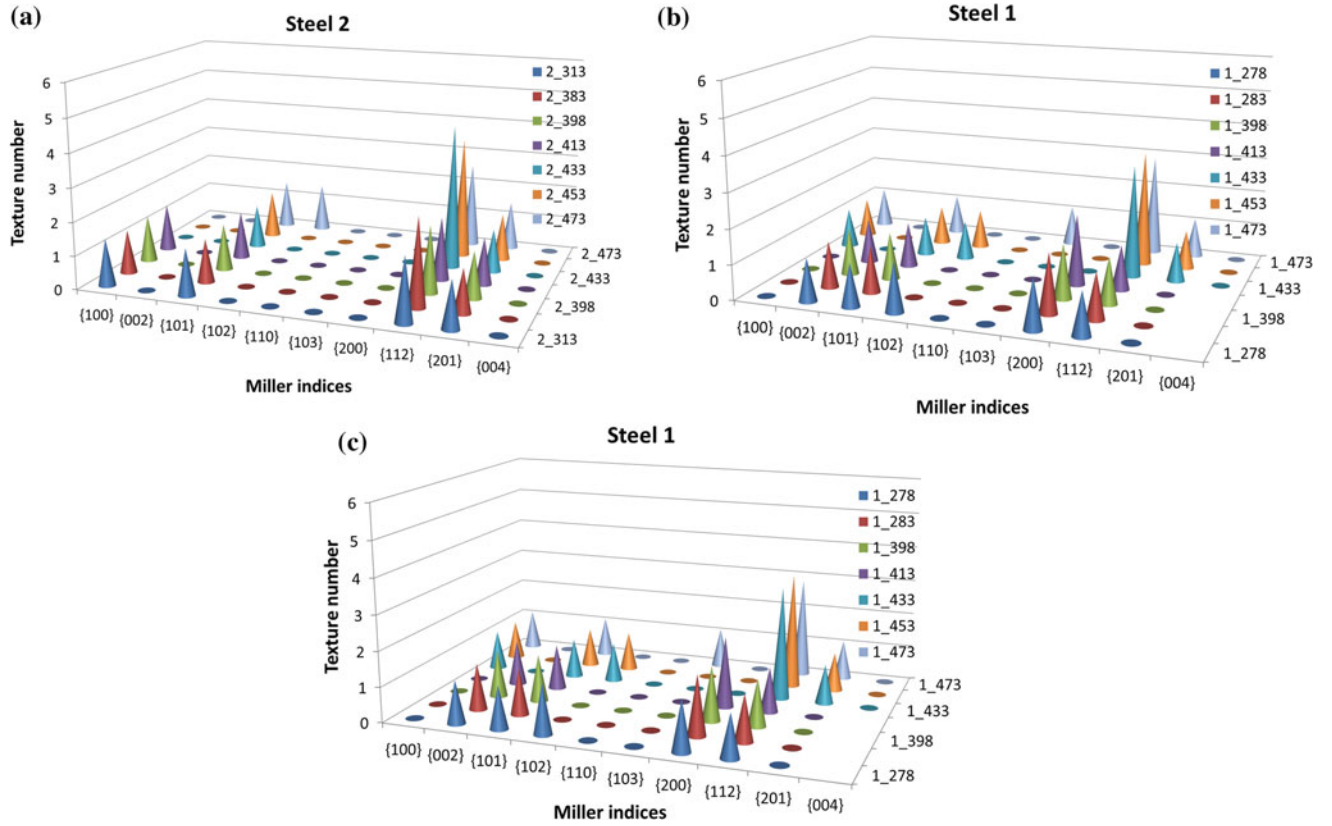


Fig. 2 Texture numbers of ϵ martensite obtained from cross section XRD spectra of **a** steel 2, **b** steel 1 and **c** steel 3 tensile tested at different temperatures [11]

ϵ martensite was isotropic at lower test temperatures, but a notable {112} texture developed at higher tensile test temperatures for all three steel types. We have already showed in our earlier paper [11] that during the DSC test the decomposition kinetic of martensites is related to this {112} texture gradient in function of test temperature. So, this temperature dependence of the {112} texture refers the different origin of ϵ martensites. According to the {112} pole figures of ϵ martensites (Fig. 3), fiber texture was developed at each case but it can be stated that the samples exhibit stronger texture at 453 K in case of all steel types.

The texture evolution of ϵ martensite with increasing test temperature was examined on microscale through EBSD orientation mapping and pole figures of Steel 1 tested at 398 and 453 K. Figures 4 and 5 show the EBSD orientation and phase maps and pole figures of γ phase and ϵ martensite. The γ {111} $\parallel$ ϵ {001} relationship is evident in both cases.

The Figs. 6, 7, 8, 9, 10 show the results of nanoscale (TEM) orientation investigations. A γ - ϵ orientations is presented of Steel 3 tested at 398 K. Note that for Steel 3 the

test temperature is above the M_s temperature (see Table 1). The microstructure is dominated by ϵ plates in which some regions of γ phase are also present (all areas of γ phase are marked). No high angle austenite grain boundary can be seen; thus, all the examined area would be one austenite grain. The ϵ plates have two main orientations. To examine the orientation relationship between γ and ϵ phases, γ [111] and ϵ [001] pole figures were taken and are shown in Fig. 6. Both γ [111] and ϵ [001] pole figures have a pole in the same orientation meaning that there is a parallel γ [111] and ϵ [001] direction. This agrees with the Kurdjumov-Sachs (K-S) orientation relationship: γ {111} $\parallel$ ϵ {001} $\parallel$ α' {110} [4, 7, 9, 10, 12].

Figure 3 XRD {112} pole figures of ϵ martensites of Steel 2, 1 and 3 tested at 398 and 453 K. CHI range of pole figures is 12° – 60°

Figure 7 shows the TEM orientation map and γ [111] and ϵ [001] pole figures of Steel 1 tensile tested at 398 K. For the composition of Steel 1, the applied test temperature is below the M_s temperature (see Table 1). The microstructure

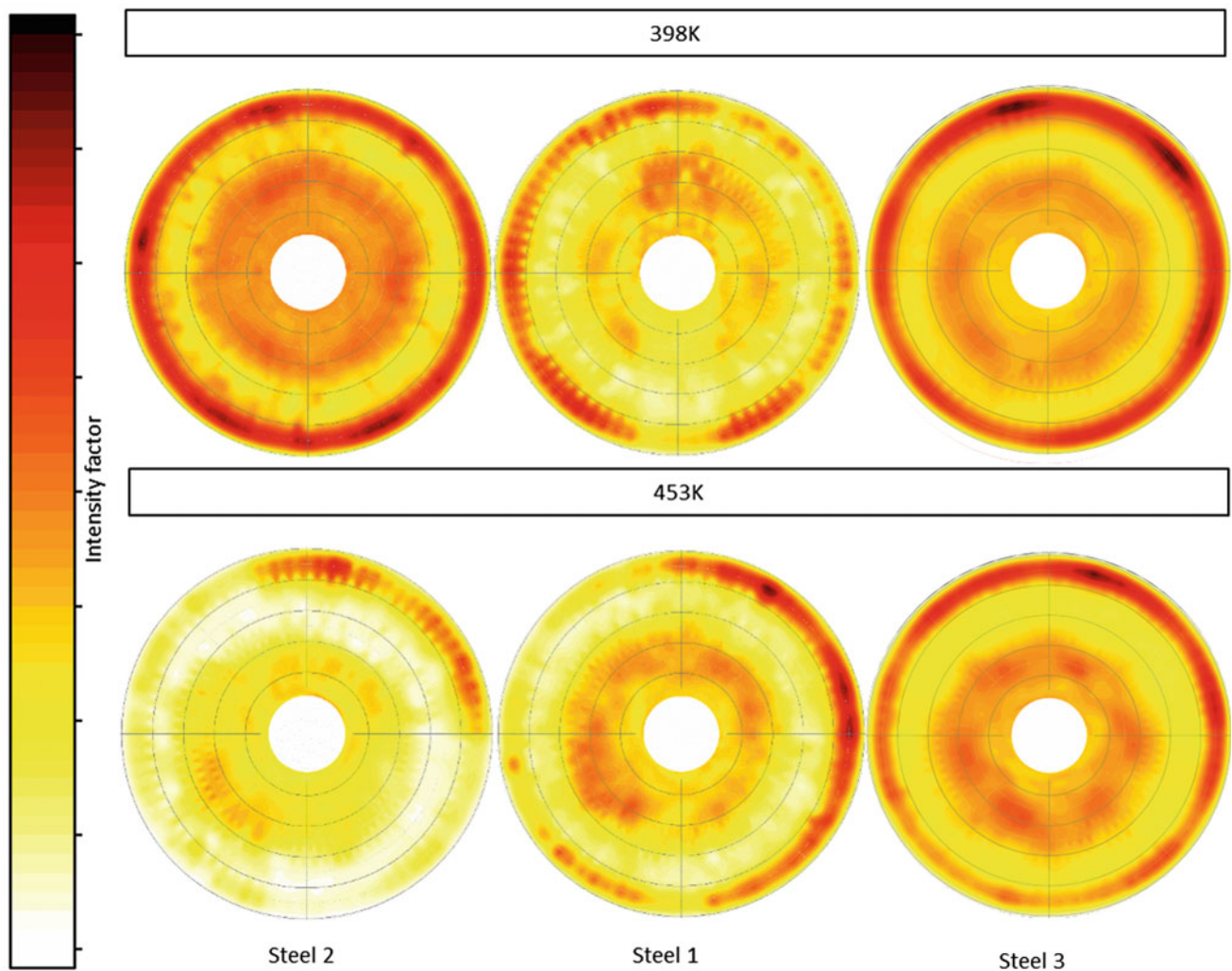


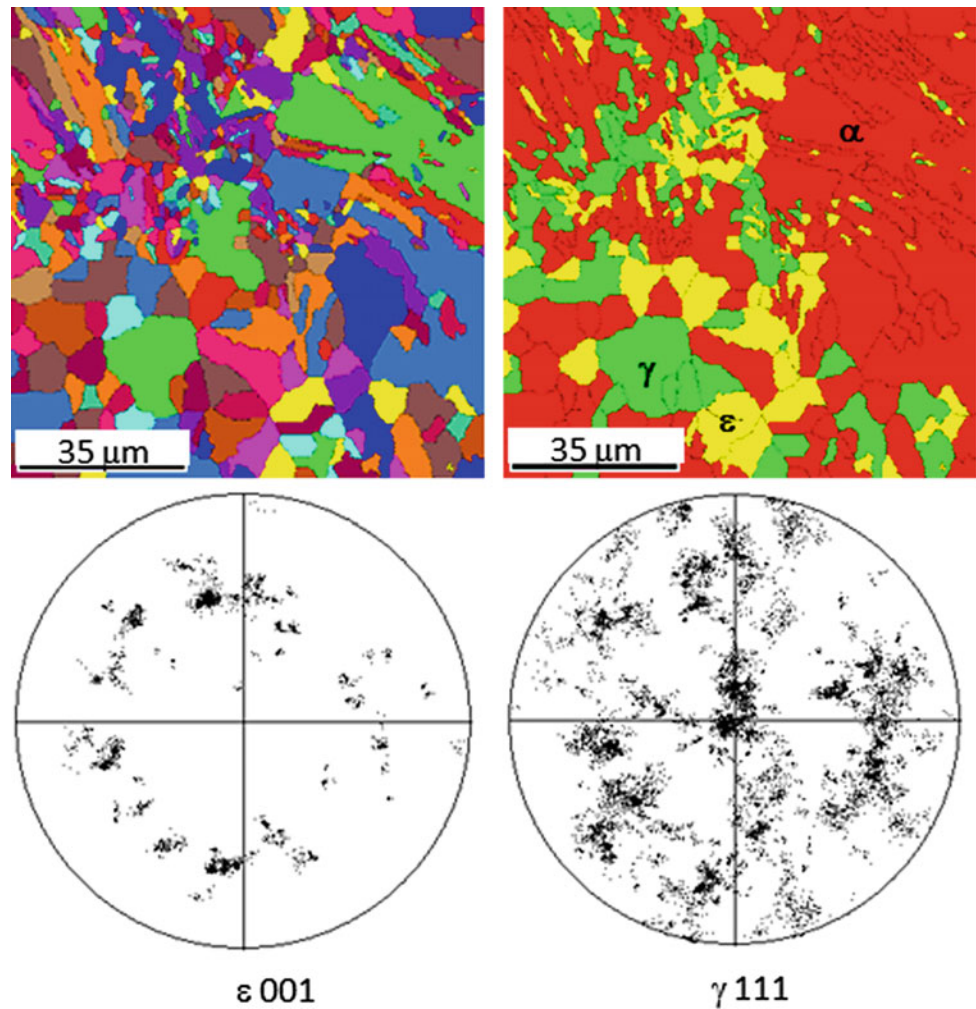
Fig. 3 XRD {112} pole figures of ϵ martensites of steel 2, 1 and 3 tested at 398 and 453 K. CHI range of pole figures is 12° – 60°

contains large regions of ϵ and α' martensite phases with some areas of γ austenite. A high angle boundary that was a grain boundary in the former austenitic structure can be seen in the upper part of the image. The locations of the γ - ϵ orientation examination are marked. The γ [111] and ϵ [001] pole figures have a common orientation meaning that there is a parallel γ [111] and ϵ [001] direction about which the γ and ϵ phases are rotated. The same correlation was found between γ and ϵ phases of a common former austenite grain during the examination of other areas as well. Furthermore, the same orientation relationship was observed between γ and ϵ phases in all the examined samples regardless of its state (as-quenched or tensile tested), test temperature or

composition. Thus, thermomechanical treatment, temperature of tensile test and variation of Cr concentration of a few wt% have no effect on the orientation relationship between γ and ϵ phases originated from the same austenite grain.

As seen in Fig. 6 all the ϵ plates found within one former austenite grain have two main orientations. Figure 8 shows ϵ [100] pole figures of two neighbouring ϵ plates having the two main orientations. The adjacent ϵ plates have a common direction in the [100] pole figure meaning that one [100] direction of the ϵ plates are parallel to each other, and the rotation axis about the examined ϵ plates are rotated relative to each other is in the direction of the common [100] direction. The angle of rotation was obtained by measuring

Fig. 4 EBSD orientation map and pole figures of steel 1 tensile tested at 398 K



the misorientation angle along a line crossing the two adjacent ε plates. The line is shown in Fig. 6. The misorientation variation of the neighbouring ε plates is shown in Fig. 8. The measured misorientation angle is highlighted. The measured misorientation angle between the neighbouring ε plates is $\sim 71^\circ$. Values of $\sim 70^\circ$, 71° misorientation angle were found to be characteristic to ε - ε relations of the same austenite grain in all the examined samples, regardless of state, tensile test temperature and composition. The measured misorientation angle of 70° - 71° which is very close to the theoretical inclination angle of the $\langle 111 \rangle$ directions in the fcc structures (70.53°). This is in agreement with the K-S relation according to which the $\gamma \leftrightarrow \varepsilon$ transformation occurs along the $\gamma \{111\}/\varepsilon \{001\}$ planes. This means that the application of thermomechanical treatment, the temperature of the thermomechanical treatment and variation of Cr concentration have no effect on the

orientation relationship between adjacent ε phases within the same austenite grain.

Due to the strain induce effect at lower testing temperature the α' martensites also formed. The Fig. 9 shows the orientation map of Steel 2 tensile tested at 398 K. The microstructure contains ε and α' martensites. The α' martensite does not have a plate-like morphology, its appearance is granular. The orientation relationship between ε martensite and α' martensite is shown by $\varepsilon [001]$ and $\alpha' [110]$ pole figures (Fig. 9). It can be seen that $\varepsilon [001]$ and $\alpha' [110]$ pole figures have a common pole orientation. Accordingly, there is a parallel $\varepsilon [001]$ and $\alpha' [110]$ directions. This relation was found to be characteristic to ε and α' martensites formed from a common austenite grain. Similarly to the previous orientation examinations, the K-S relation was found to be valid for the ε martensite and stress induced α' martensite as well.

Fig. 5 EBSD orientation map and pole figures of steel 1 tensile tested at 453 K

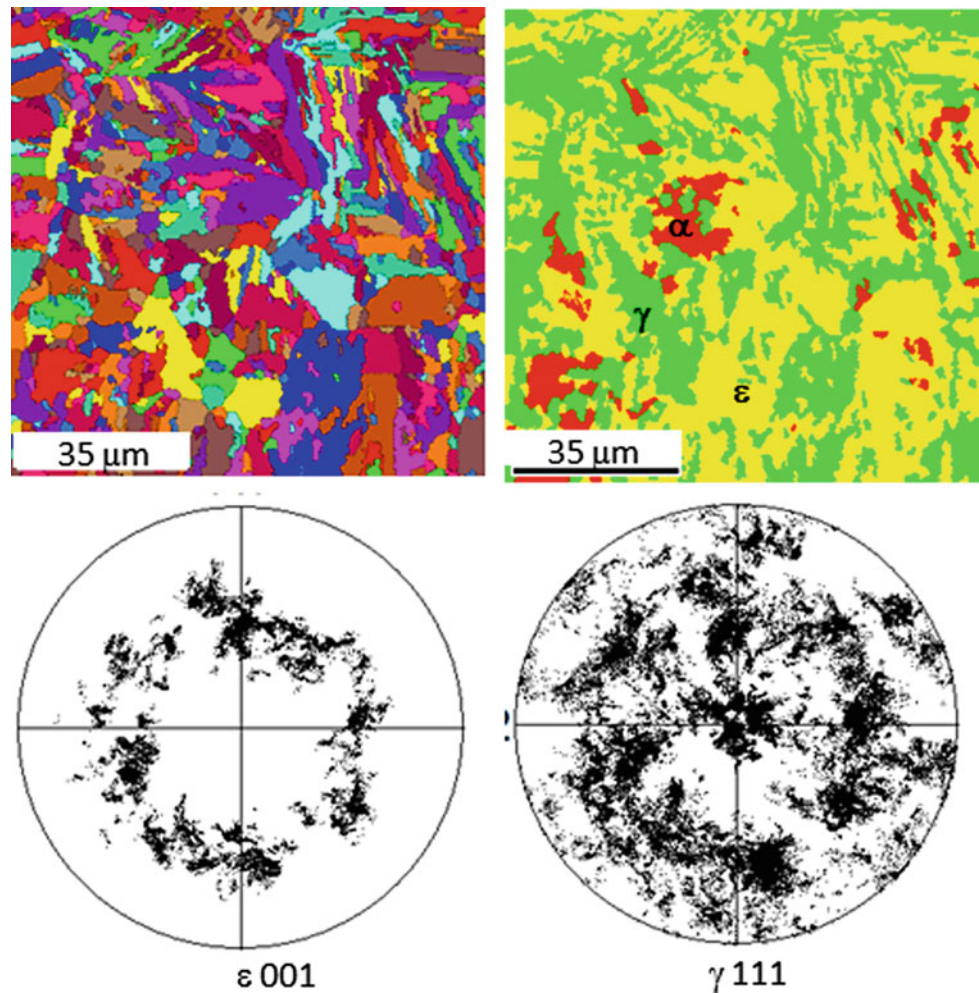
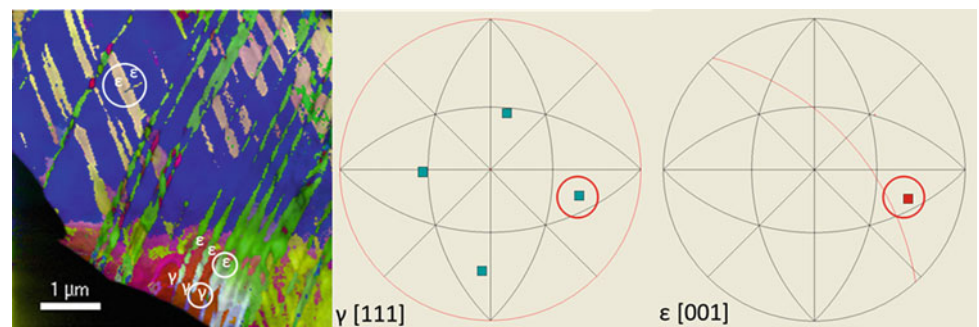


Fig. 6 TEM orientation map and pole figures of steel 3 tensile tested at 398 K



The γ - α' orientations were also investigated. Figure 10 shows the microstructure of the Steel 1 tested at 398 K. Plates of α' martensite and some regions of γ austenite are present in the ϵ matrix. A high angle grain boundary can be seen passing through horizontally in the middle that was an austenite grain boundary. The examination of the γ - α'

orientation was carried out by measuring pole figures in γ and α' phases within the same former austenite grain (marked). As seen in Fig. 10, γ [111] and α' [110] poles are in the same direction meaning that the γ and α' phases have a parallel γ [111] and α' [110] direction. Thus, the K-S relation was confirmed by the γ - α' relationship as well.

Fig. 7 TEM orientation map and pole figures of steel 1 tensile tested at 398 K

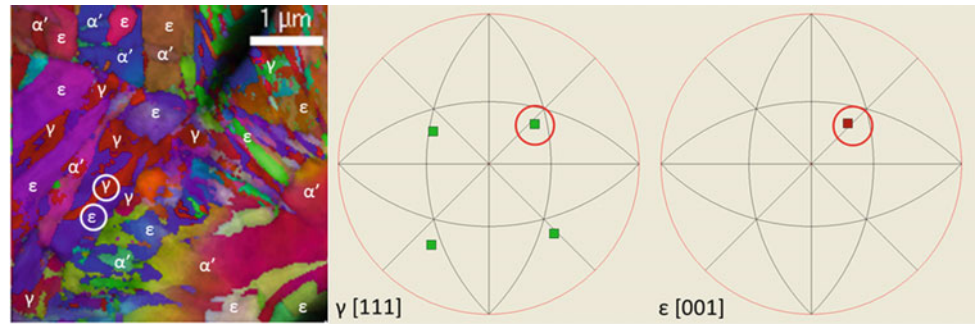


Fig. 8 ϵ [001] pole figures and misorientation angle between two adjacent ϵ plates of steel 3 tensile tested at 398 K

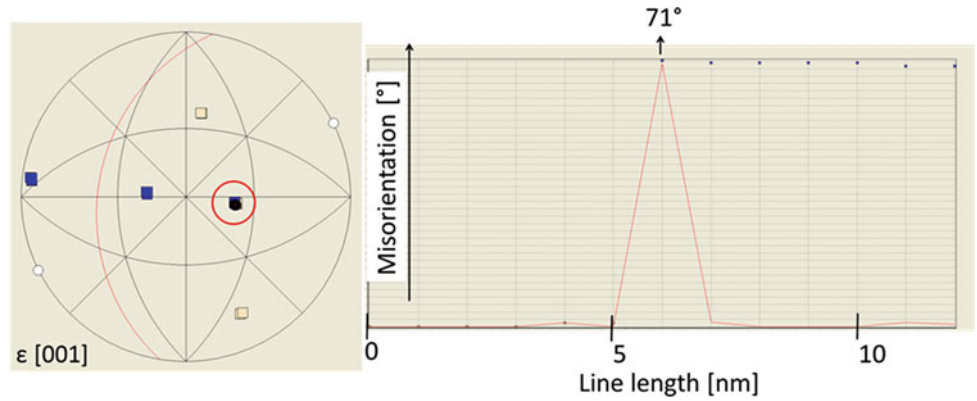


Fig. 9 Orientation map of steel 2 tensile tested at 398 K and the ϵ [001] and α' [011] pole figures

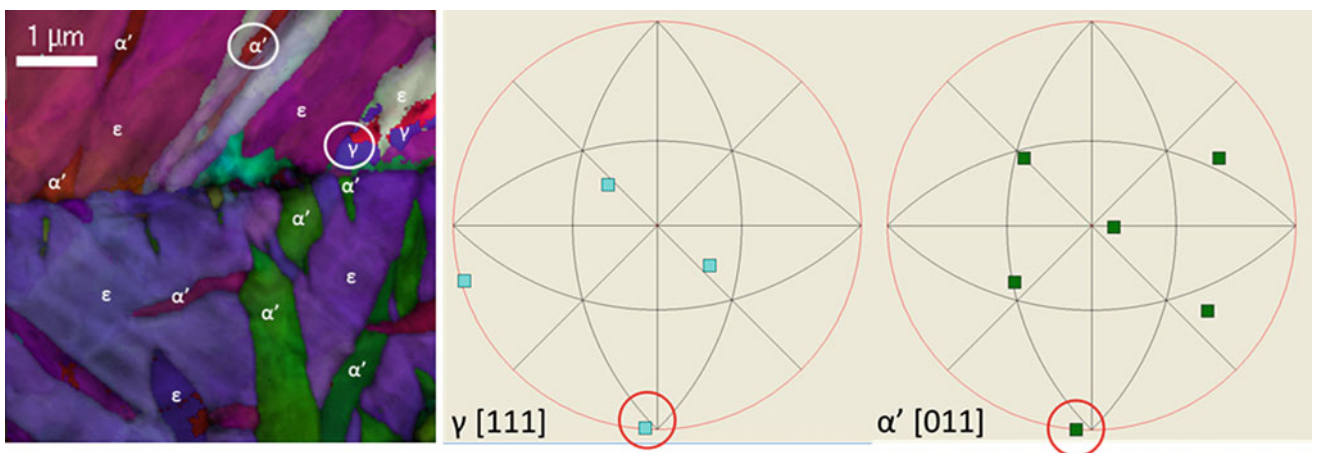
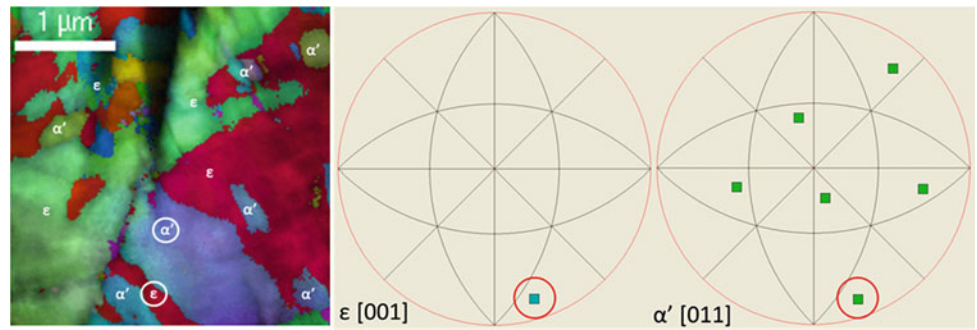


Fig. 10 Orientation map of steel 1 tensile tested at 398 K and the γ [111] and α' [011] pole figures with the locations of γ - α' orientation examination

Conclusions

Orientation relationships were investigated between γ austenite, ε martensite and α' martensite in TWIP/TRIP steel samples having different Cr content after tensile tests carried out at different temperatures using XRD, EBSD and TEM orientation mapping. Within the same former austenite grain, the Kurdjumov-Sachs (K-S) orientation relationship ($\gamma \{111\} \parallel \varepsilon \{001\} \parallel \alpha' \{110\}$) was confirmed between adjacent γ - ε , ε - α' and γ - α' phases in all three examined steels (Cr content: 0, 2.26, 6.12 wt%), in as-quenched and tensile tested samples regardless of whether tensile test was carried out above or below the M_s temperature. Previous XRD texture investigations revealed that the texture number related to the $\varepsilon \{112\}$ planes increase with tensile test temperature. Now, it was shown that the $\varepsilon \{112\}$ has a fiber texture which is stronger at higher test temperatures. The $\{112\}$ fiber texture could only be observed on the macroscopic scale.

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