Microstructures and textures comparison of conventional and high Niobium API 5L X 80 line pipe steel using EBSD

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The development of microstructure and crystallographic texture of conventional and high Niobium API 5L X80 line pipe steels has been studied using Electron Backscattered Diffraction (EBSD) technique. The selective use of micro-alloying elements like niobium in alloy design, increases the recrystallization stop temperature, which facilitates rolling at higher temperatures compared to conventional thermo-mechanical processing. The measurement tools available with EBSD, such as image quality values and precise grain boundary misorientation angles, provide new approaches to characterize and compare both microstructures. The texture analysis, in addition, shows high intensification of {112}<110>, {554}<255> and rotated cube {001}<110> for both processes. However, the Goss {110}<001> and the rotated Goss {110}<110> has been only observed in the conventional processing which is a sign that the austenite was recrystallized prior to transformation. This shows the ease of processing experienced with High Temperature Processing (HTP) chemistry compared to the conventional one.

Keywords: API X80 - Steel - EBSD - Microstructure - Texture - Image Quality - Misorientation distribution

INTRODUCTION

The trend towards increased pipeline operating pressures while keeping steel costs low has resulted in higher strength steel such as API X70 (483 MPa) and X80 (550 MPa) becoming place. API X80 has been traditionally produced using C-Mn-Mo based highly dislocated ferritic microstructure. The cost of molybdenum (Mo) was of concern and a need for an alternative cost-effective alloy design that can create similar microstructures and hence properties. The High Temperature Processing (HTP) concept using an alloy design of mainly C-Mn with Nb has allow for thermo-mechanical rolling at higher temperatures with the ability to produce the required properties. This is due to the fact that solute Nb has the ability to retard recrystallization which results in finer ferritic microstructure after austenite-ferrite transformation [1].
The understanding of such products is important to identify the appropriate alloys and processing parameters for better control of mechanical properties. The development of Electron Back-Scatter Diffraction (EBSD) method offers an innovative way to analyze such microstructures more effectively and efficiently than the traditional light microscopy or Scanning Electron Microscopy (SEM). The extensive measurement tools available with EBSD, such as image quality values and precise grain boundary misorientation angles, provide new approaches to characterize microstructures [2,3,4].

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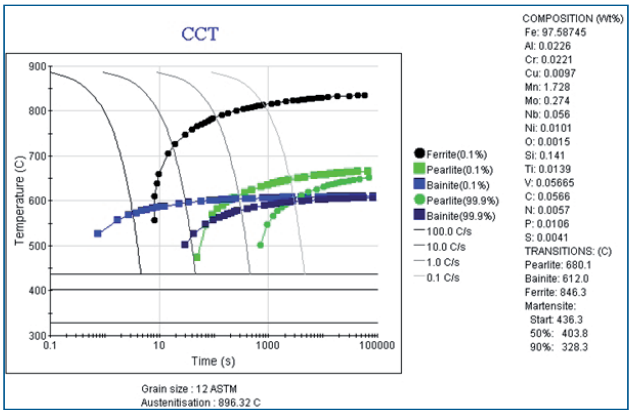
Material	C	Mn	Nb	Cr+Ni+V	Others
API X80 Conv.	0.04-0.07	1.75-1.85	0.05-0.06	≤ 0.3	Ti, Nb, Cr and Mo
API X80 HTP	≤ 0.07	1.50-1.70	0.07-0.10	≤ 0.3	Ti, Nb and Cr

Tab. 1 - Chemical composition of the investigated steels.

Tab. 1 - Composizione chimica degli acciai esaminati.

MATERIALS AND PROCESSING

Two steel chemistries, conventional and HTP, were used in this study for the production of API X80 and are shown in Table 1. Both chemistries are low carbon Ti-Nb-V added high strength low alloy (HSLA) steels but with the addition of Mo in the conventional one and higher Nb content in the HTP design.
The HTP alloy design yielded CCT curves (Figure1b) that showed the required highly dislocated ferritic microstructure could be developed with accelerated cooling rates of 15-20 °C/sec.
The rolling parameters were finalized for thickness range 13-16mm. Both chemistries were reheated upto 1200 °C, finish rolled at 820 °C for conventional chemistry and 860°C for HTP chemistry, and coiled at 600 °C to allow slow cooling to room temperature.



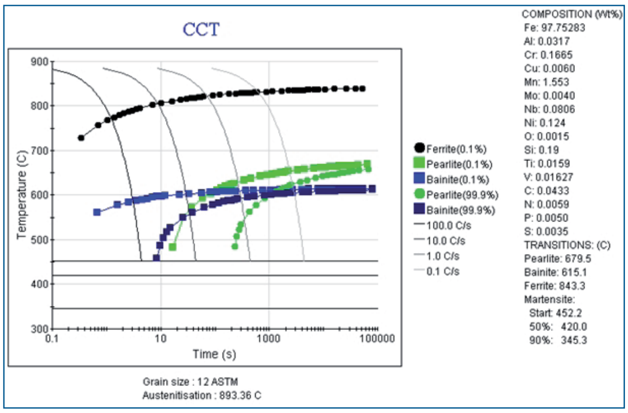


Fig. 1 - CCT curves for API X80 with (a) Conventional, and (b) HTP alloy designs.

Fig. 1 - Curve CCT per l'acciaio API X80 (a) convenzionale, e (b) HTP con alligazione.

Samples from both trials in two orientations, transverse and diagonal to rolling direction, were prepared for tensile tests, drop weight tear tests (DWTT), impact tests (-10°C and -40 °C) and hardness tests. Ductile shear area resulted after the DWTT was calculated as per the evaluation procedure described in the API specification [5].

Samples have also been collected from both steels for Electron Back-Scatter Diffraction (ESBD) analysis. The samples were cut using diamond precision cutter to minimize the surface deformation during cutting. Then, the specimens were ground and polished using automatic grinder/polisher machine.

Test	Conventional X80		HTP X80	
	Transverse	Diagonal	Transverse	Diagonal
YS (MPa)	606	560	645	595
TS (MPa)	718	708	710	690
El. (%)	33	36	33	35
DWTT @ 0°C (% SA)	> 90	> 85	> 95	> 85
CVN Impact @ -10°C (J)	277	269	237	247
CVN Impact @ -40°C (J)	260	212	195	240
Hardness (HV)	234		226	

Tab. 2 - Mechanical properties of the investigated steels

Tab. 2 – Proprietà meccaniche degli acciai esaminati.

MECHANICAL TESTING

Table 2 shows the mechanical properties for both steels, including yield and tensile strengths, SA% measured at the fractured surfaces of the DWTT specimens, impact tests done at -10 and -40 °C, and hardness values. The results indicate that both steels have passed the minimum requirements for X80 steel grade. The yield strength values were higher for HTP steel while the tensile strength values were almost similar. The table also indicates that both steels exceeded the 85% SA criterion. The impact tests show high values even at low temperatures of -40 °C.

MICROSTRUCTURE INVESTIGATION USING EBSD

The Electron Back-Scatter Diffraction (EBSD) technique offers an innovative way to analyze the microstructures more effectively and efficiently than the traditional light microscopy or Scanning Electron Microscopy (SEM). Figure 2a and c show EBSD maps (Euler color) of the two investigated steels. More structural bands can be observed on the conventional chemistry when compared to HTP one. The grain boundary maps are shown in Figure 2b and d for both steels. The misorientation angles are divided into low and high angle boundaries using the traditional terminology. Low angle grain boundaries (LAGB) are those with misorientation angle less than 15° (red) while the high angle grain boundaries (HAGB) have misorientation angles greater than 15° (blue). The HAGB to LAGB length ratios are 2.11 and 2.23 for the conventional and HTP steels, respectively. This indicates that both steels are having almost similar microstructures. Figure 3 shows the average grain sizes for both steels using optical and EBSD technique where a slight increase of grain size is observed with the EBSD technique.

GRAIN BOUNDARY INVESTIGATION

The misorientation distribution may offer a different approach to characterize the complex ferrite microstructures. Figure 4 shows a comparison between the misorientation angle distribution of the two investigated steels. The major peaks positions are almost identical for both steels located at 30, 45, and 60°. The misorientation distribution is a characteristic for the transformation mode generating the steel microstructure. The existence of substructure produces a minor peak below 10°. The maximum observed close to 60° is probably related to twining process indicating that an athermal transformation has taken place. The overlaps in the misorientation profiles of lower bainite and martensite (associated with misorientation peak at around 60°), as well as of upper bainite and acicu-

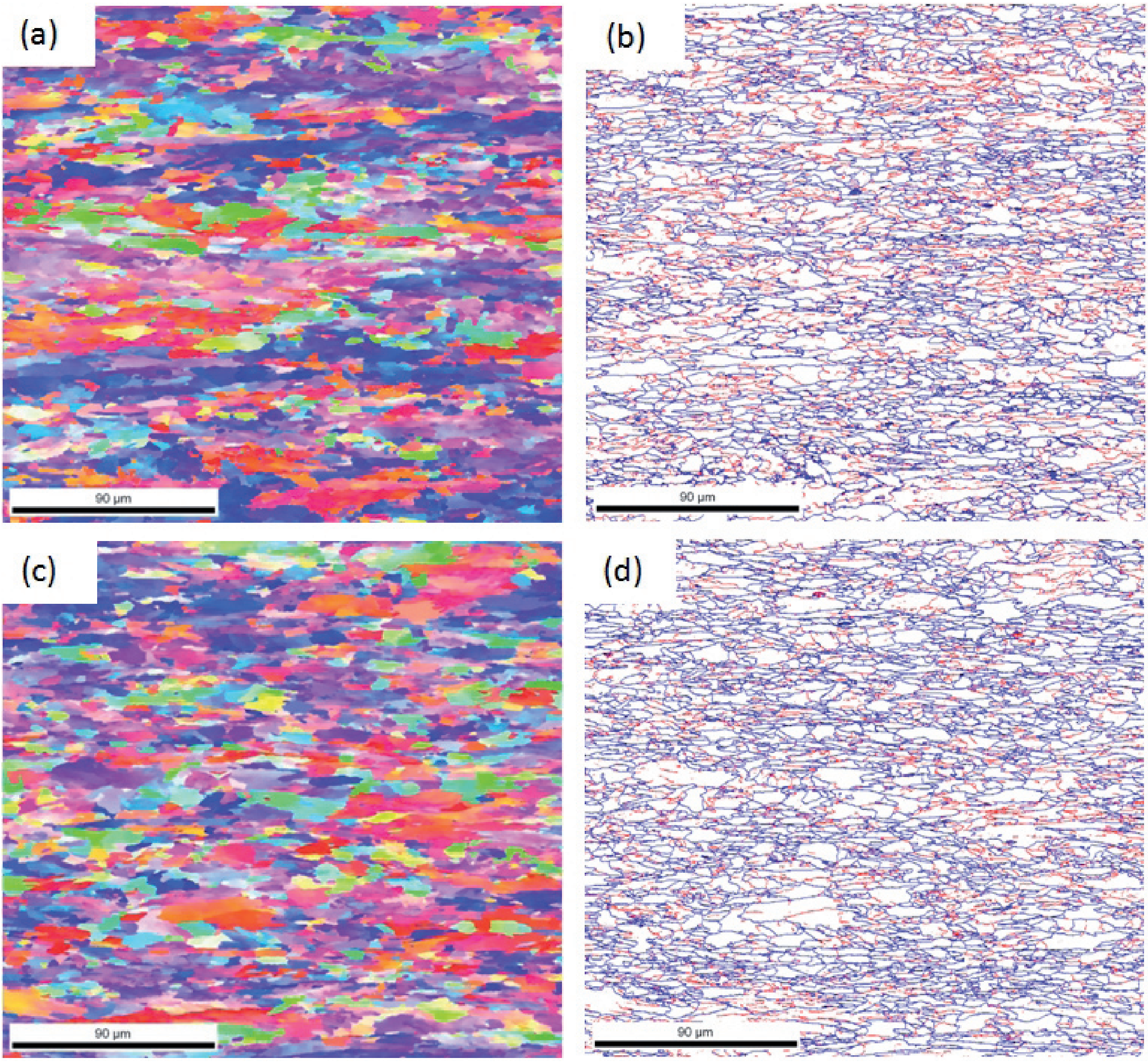


Fig. 2 - EBSD maps (Euler color) and grain boundary maps for a,b) Conventional steel, and c,d) HTP steel.

Fig. 2 - Mappe EBSD (colori Eulero) e mappe dei bordi grano per a,b) acciaio convenzionale, e c,d) acciaio HTP

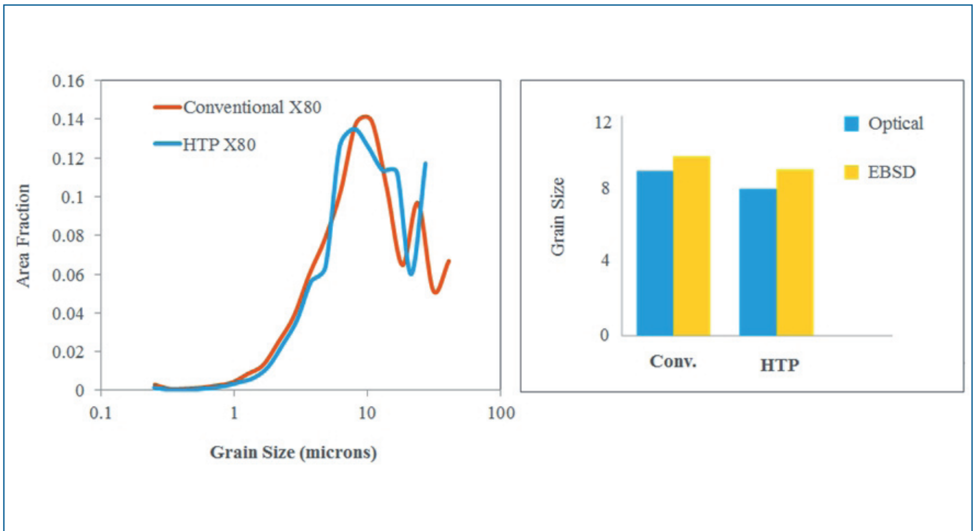


Fig. 3 - (a) Grain sizes measurements for both steels using EBSD, and (b) Comparison of average grain sizes measured by optical and EBSD techniques for both steels.

Fig. 3 – Misure delle dimensioni dei grani per entrambi gli acciai mediante EBSD e (b) confronto fra dimensioni medie dei grani misurate mediante microscopia ottica ed EBSD per entrambi gli acciai.

lar ferrite (peak at around 45°) restrict the application of this technique to define the appropriate misorientation ranges for different ferrite morphologies.

IMAGE QUALITY (IQ) ANALYSIS

Examples of the IQ distributions for the two investigated steels can be seen in Figure 5. Normalization of IQ values has been done to minimize the effects of experimental set-up and specimen preparation on pattern quality. In addition, it brings values which are quantitatively comparable. The normalized IQ values have been calculated using Eq. (1).

$$IQ_{Normalized} = \frac{IQ_{Initial} - IQ_{Min}}{IQ_{Max} - IQ_{Min}} X100 \quad (1)$$

Where $IQ_{Initial}$ is the absolute IQ value gained directly from experiment, and IQ_{Max} and IQ_{Min} are, respectively, the maximum and minimum IQ values in the scanning set.
Since there is no point of interest in this work to study the properties of the grain boundaries, their pixels have been identified and filtered out (Figure 5b). Removing the grain boundary (GB) pixels has the effect of reducing an apparent shoulder typically seen on the left side of the major peak on the high IQ side of the distribution, without affecting the major peak positions as shown by comparing Figures 5a and 5b. A slight increase in the IQ values for all major peaks can be observed after GB removal.
It can be clearly seen that the sharpness of the peaks is almost identical for both steels. This can be attributed to the fact that the microstructure, and the lattice defect density, of both steels are almost similar. Although, a slightly higher IQ value for the HTP X80 peak can be observed, this can be due to the effect of the image processing conditions such as accelerating voltage and beam current.

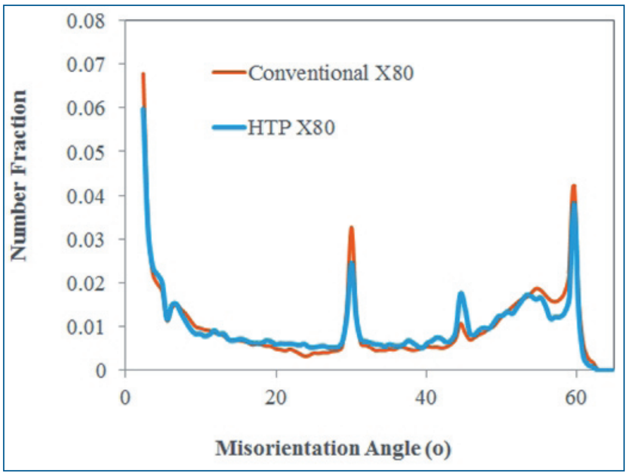


Fig. 4 - Distribution of ferrite grain boundaries misorientations for both steel investigated.

Fig. 4 – Distribuzione della disorientazione dei bordo grano della ferrite per entrambi gli acciai esaminati.

TEXTURE MEASUREMENT

The transformation texture for each of the two steels investigated were measured and can be seen in Figure 6 using ϕ2 = 45° section of the Euler space. The texture analysis shows high intensification of {112}<110>, {554}<255> and rotated cube {001}<110> for both processes. However, the Goss {110}<001> and the rotated Goss {110}<110> has been only observed in the conventional processing which is a sign that the austenite was recrystallized prior to transformation [6]. This shows the ease of processing experienced with High Temperature Processing (HTP) chemistry compared to the conventional one.

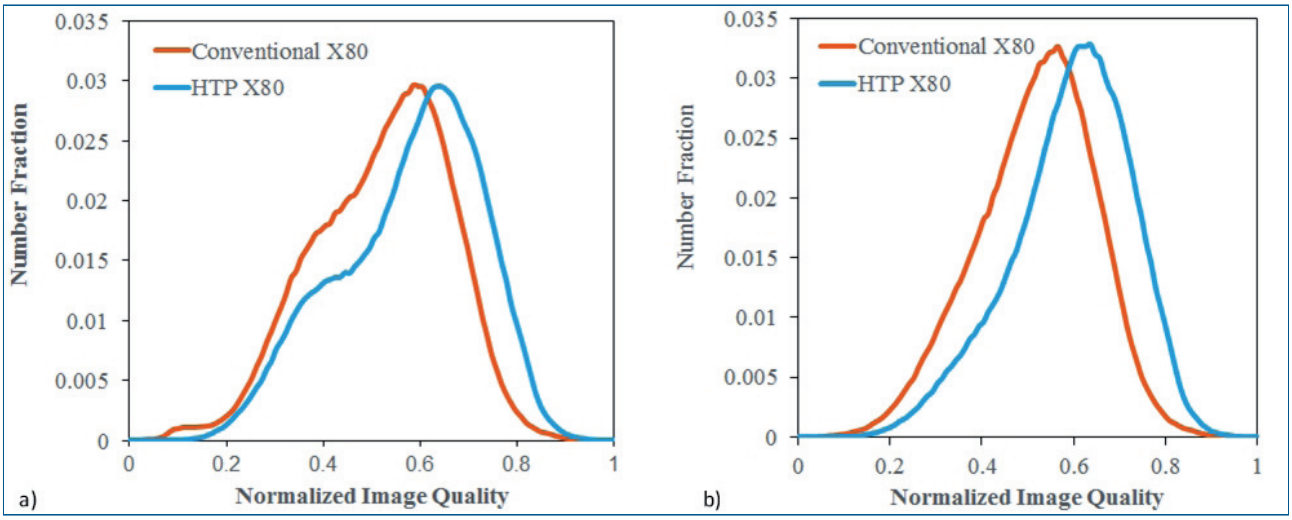


Fig. 5 - (a) Normalized IQ distribution illustrating different peak locations for the two investigated steels, (b) same with grain boundary pixels removed.

Fig. 5 - (a) Distribuzione IQ normalizzata che mostra differenti posizioni dei picchi per i due acciai esaminati, (b) la stessa con eliminazione dei pixel dei bordi grano.

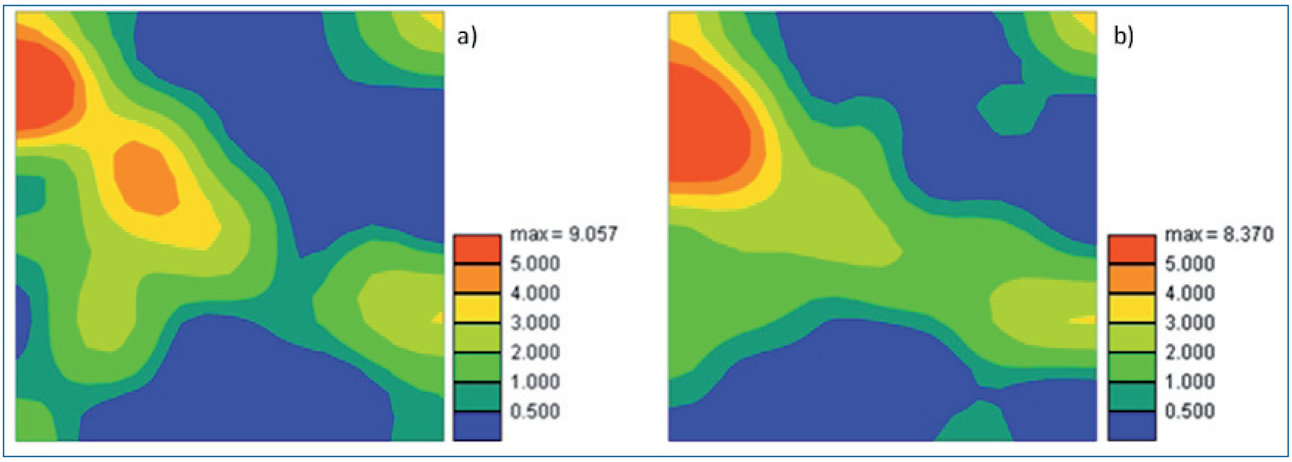


Fig. 6 - Textures of the final ferritic structure (ODF sections at $\phi 2=45^o$) of (a) conventional API X80, and (b) HTP X80 steels.

Fig. 6 - Tessiture della struttura ferritica finale (sezioni ODF a $\phi 2=45^o$) di (a) acciaio convenzionale API X80, e (b) acciaio HTP X80.

CONCLUSIONS

Conventional and high Niobium chemistry designs for API 5L X80 line pipe steels, developed successfully at SABIC, has been studied using Electron Backscattered Diffraction (EBSD) technique. This technique offers a new and reliable approach to characterize the complex and varying microstructures present in the investigated steel such as image quality values and grain boundary misorientation angles. The texture measurement has confirmed the ease of processing experienced with High Temperature Processing (HTP) chemistry compared to the conventional one.

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Confronto mediante EBSD fra microstrutture e tessiture di acciaio convenzionale e di acciaio per tubazioni ad alto tenore di niobio API 5L X80

Parole chiave: Acciaio – Deformazioni plastiche - Laminazione

Nel presente lavoro è stato studiato lo sviluppo di microstruttura e tessitura cristallografica di acciaio convenzionale e di acciaio per tubazioni ad alto tenore di niobio API 5L X80 usando la tecnica di diffrazione di elettroni retrodiffusi (EBSD). L'uso selettivo nella lega di elementi microleganti, come il niobio, aumenta la temperatura di arresto della ricristallizzazione, facilitando la laminazione a temperature più elevate rispetto al trattamento termo-meccanico convenzionale. Gli strumenti di misura a disposizione con EBSD, come ad esempio i valori di qualità dell'immagine e gli angoli di disorientazione dei bordi di grano, offrono nuovi approcci per caratterizzare e confrontare entrambe le microstrutture.
L'analisi della tessitura, inoltre, mostra un'alta intensificazione degli indici {112} <110>, {554} <255> e del cubico ruotato {001} <110> per entrambi i processi.
Tuttavia, gli indici Goss {110} <001> e Goss ruotato {110} <110> sono stati osservati solo nel trattamento convenzionale e ciò indica che l'austenite è stata ricristallizzata prima della trasformazione. Questo dimostra la facilità di lavorazione con il metodo High Temperature Processing (HTP) rispetto al processo convenzionale.