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Original Article

Texture, microstructure and anisotropic properties of IF-steels with different additions of titanium, niobium and phosphorus☆

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ABSTRACT

Interstitial free steels (IF steels) find wide application in the automotive industry, due to their excellent drawability. This work aims to study the microstructural and mechanical properties variations that might occur in an IF steel, when amounts of titanium, niobium and phosphorus are added to a specific alloy composition. The goal is to address how the insertion of such elements could provide excellent deep drawing applications without compromising the steel mechanical strength. Mechanical testing was performed in different samples to determine the steel anisotropy and compare those to the calculated crystallographic texture obtained by X-ray diffraction analysis. Scanning transmission electron microscopy (STEM) was also performed to correlate the properties achieved by the alloying elements to the microstructure of the steel rolled sheets.

1. Introduction

Interstitial free steels (IF steels) are a class of steels that are designed to have a minimal content of interstitial elements within their matrix, to achieve high ductility. Their industrial production started in the 1970s, with the main objective of fabricating steels with greater formability for deep drawing applications without compromising its costs. Today, this class of steels is fabricated in many parts of the world, due to the demand from the automotive industry for the manufacturing of automotive panels [1].

IF steels have a ferritic matrix with body centered cubic (BCC) crystal structure and a composition of ultra-low carbon and nitrogen content (less than 0.003% by weight). To achieve very low levels of interstitial elements, modern vacuum degassing (RH) techniques are used during the fabrication process. The addition of microalloying elements such as titanium

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and/or niobium, can lead to the formation of precipitates (carbides and nitrides) so that the ferritic matrix of the IF steel is practically free of carbon and nitrogen interstitial atoms. However, the greater acquired drawability is usually accompanied by loss in mechanical strength, which became the great challenge in the development of these steels [2]. When exceptional strength is necessary, addition of other alloying elements, such as phosphorus, silicon and manganese, is incorporated to the IF steels, to promote solid solution hardening and thus increasing their resistance (high strength IF steel (IF-HS)).

Recent works [3–5] have been discussing the effects of phosphorus additions on texture evolution and the effects of process parameters in the IF steel microstructure. However, the exact behavior of the transformations in IF steels with phosphorus additions still requires further and deeper investigations.

Many works have investigated the addition of only titanium (Ti), titanium with phosphorus (Ti + P) and only niobium (Nb) to IF steels, but very little has been studied regarding the triple addition of Ti, Nb and P. A work by Takechi [2] showed that niobium has a better solubility than titanium, suggesting that the IF-Nb steel might lead to a better resistance to fragilization than the IF-Ti. Thus, the combination of both elements could produce an IF-steel with improved properties. However, the effect of the combination of titanium, niobium and phosphorus to the steel properties is not easily found in current literature.

This work aims to study the microstructural and mechanical properties variations that might occur in an IF steel, when amounts of titanium, niobium and phosphorus are added to a specific alloy composition. The goal is to address how the insertion of such elements could provide excellent drawability without compromising the steel mechanical strength. It is, therefore, crucial that {111} crystal texture is preserved after all the thermomechanical processing is completed. To verify this condition, mechanical testing was performed in different samples to determine the steel anisotropy and compare those to the calculated crystallographic texture obtained by X-ray diffraction analysis. Scanning transmission electron microscopy (STEM) was also performed to correlate the properties achieved by the alloying elements to the microstructure of the steel rolled sheets.

2. Experimental procedures

2.1. *Chemical composition of steel alloys*

Three distinct types of IF steels were prepared, regarding the alloying elements that were incorporated: IF-Ti (titanium addition); IF-TiNb (titanium and niobium addition); and IF-TiNbP (titanium, niobium and phosphorus addition). Their specific chemical composition is presented in Table 1.

2.2. *Rolling simulations*

The rolling has been carried out to simulate the industrial processing performed in IF steels used as automotive parts. The samples were hot and cold rolled from 80 × 50 × 25 mm to 1 mm thick-sheets. The specific steps of all processes are described elsewhere [6].

2.3. *Tensile tests*

Tensile tests were performed in each alloy for acquiring data related to the material mechanical parameters such as yield strength, tensile strength and elongation. Each different steel (IF-Ti, IF-TiNb and IF-TiNbP) had samples obtained from three different directions in relation to the rolling directions, i.e., samples were withdrawn from the parallel (0°), 45° and 90° direction regarding the initial rolling direction. Specimens for tensile tests were prepared via electrical discharge machine (EDM wire cutting), with a geometry according to ASTM E8/E8M-11 specifications. The uniaxial tensile test was performed by an EMIC mechanical testing machine, with a capacity of 98 MN and at ambient temperature with a strain rate of $10^{-3}\,s^{-1}$.

2.4. *Scanning and transmission electron microscopy (STEM)*

Morphology analysis of the precipitates present in the microstructure of the three steel types was carried out by a "FEI Titan G2 80-200" scanning transmission electron microscope (STEM) equipped with a Bruker ChemiSTEM/Espirit Energy Dispersive X-ray Spectrometer (EDS). Samples were cut into 3 mm diameter discs with 200 μm thickness, mechanically and electrolytically polished in a solution of 9 vol% of perchloric acid and 91 vol% of glacial acetic acid, using a voltage of 25 V and at the temperature of 15 °C ± 1 °C. Both transmission electron microscopy (TEM) and scanning transmission electron microscopy (STEM) modes were used for the sample assessment.

2.5. *Crystallographic texture analysis*

Crystallographic texture measurements were performed using a PANalytical X-ray diffractometer, with cobalt radiation (wavelength 1789 Å) and operating at voltages of 40 kV and 45 mA current. Samples were prepared in dimensions of 20 × 20 mm and both the surface and half-thickness were analyzed.

Table 1 – Chemical composition of the IF-steel and its alloying elements (wt%).

IF steel	C	N	Nb	Ti	P	Al	S	Mn	Si
IF-Ti	0.0021	0.0025	0.0018	0.0820	0.0099	0.0346	0.0063	0.1250	0.0180
IF-TiNb	0.0022	0.0038	0.0160	0.0700	0.0083	0.0361	0.0093	0.1120	0.0710
IF-TiNbP	0.0022	0.0037	0.0288	0.0334	0.0298	0.0384	0.0071	0.1750	0.0500

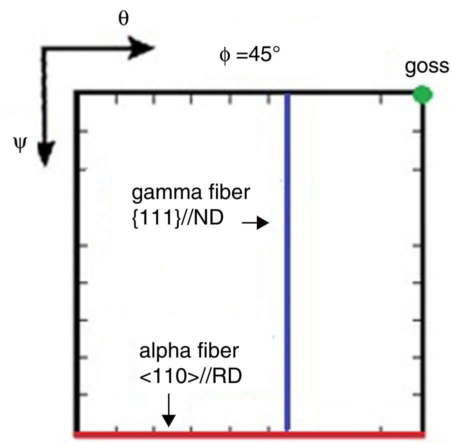

Fig. 1 – Textures components for (BCC) materials at the $\phi = 45°$ section (Roe's notation).

IF steels have body-centered cubic crystal structure (BCC), therefore, the interest diffraction planes to be analyzed, regarding crystallographic texture, are (110), (200) and (211) [7]. In terms of texture components, the most important orientations to be addressed are the ones known as alpha fiber and gamma fiber. Alpha fiber rises from $\{hkl\}\langle 110\rangle$ components when directions $\langle 110\rangle$ are parallel to the rolling direction (RD) and gamma fiber when component $\{111\}\langle uvw\rangle$ has its $\langle 111\rangle$ direction coincident with the normal direction of the rolled sheet surface (ND).

The results of the crystallographic texture analysis are illustrated by the orientation distribution functions (ODFs), in which the intensities of the texture components as a function of the angles of rotation (Euler) can be observed. The ODFs figures were generated by the popLA program (preferred orientation package – Los Alamos) using Roe notation [8] and were presented in constant $\phi = 45°$ sections, where alpha and gamma fibers can be fully observed (Fig. 1).

3. Results

3.1. Mechanical properties

Fig. 2 depicts the curves obtained for IF-Ti, IF-TiNb and IF-TiNbP steels. Apart from the IF-TiNbP steel, which presented a discontinuous yielding effect, all the other steels showed similar behavior during the tensile tests. IF-TiNb steel though, revealed the lowest value for fracture limit.

The strain-hardening coefficient (n), the normal anisotropy (r_m) and the planar anisotropy (Δr) of each steel were calculated according to the values obtained for yield strength, tensile strength and elongation in tensile tests [9]. These results are shown in Table 2.

3.2. Microstructure

Microstructure investigation was performed in order to reveal the type of precipitates that could be present in the IF steels. For IF-Ti (Fig. 3a) steel, precipitates of type TiS and TiN were observed, while in the IF-TiNb (Fig. 3b), precipitates of NbC, $Ti_4C_2S_2$. IF-TiNbP (Fig. 3c) also evidenced the existence of a precipitate FeTiP. The respective EDS are presented below each micrograph.

3.3. Crystallographic texture

Fig. 4 shows the constant $\phi = 45°$ sections of the ODFs of the rolled sheets. Fig. 4a (surface) and 4d (half thickness) are concerned to the textures components from IF-Ti steel. Fig. 4b (surface) and 4e (half thickness) present textures from IF-TiNb and Fig. 4c (surface) and 4f (half thickness) textures from IF-TiNbP.

For each ODFs, an intensity scale is presented to illustrate the variations occurring in the Euler angles (Roe's notation). The schematic presented in Fig. 1 indicates the location of the main characteristic fibers and components of IF steels. The schematic figure can be used for comparison to the results presented in Fig. 4a–f.

4. Discussion

In order to achieve the desirable structural and mechanical characteristics required for good drawability, IF steels must present specific values of anisotropy that can assure that their thickness will be preserved during deformation and their mechanical resistance will not be compromised.

The steel anisotropy is a consequence of the crystallographic texture developed after the rolling process. IF steels should present high intensity values for gamma fiber and low intensity values for alpha fiber [7], i.e., the results for normal and planar anisotropy (r_m and Δr) should vary accordingly. As reported by literature, values of normal anisotropy should be

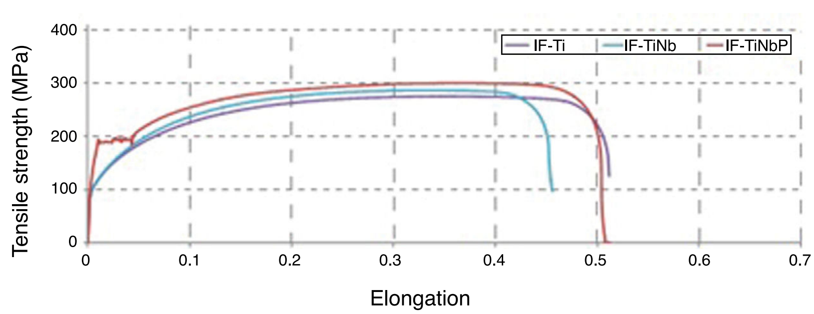

Fig. 2 – Stress x elongation curves in steels after annealing.

Table 2 – Results of uniaxial tensile tests and calculated anisotropy parameters.

IF steel	r_m	Δr	n	Yield strength (MPa)	Tensile strength (MPa)	Elongation
IF-Ti	1.92	1.98	0.296	105.19	260.21	0.41
IF-TiNb	1.16	1.11	0.304	120.84	278.40	0.42
IF-TiNbP	1.28	−0.09	0.299	162.09	322.54	0.34

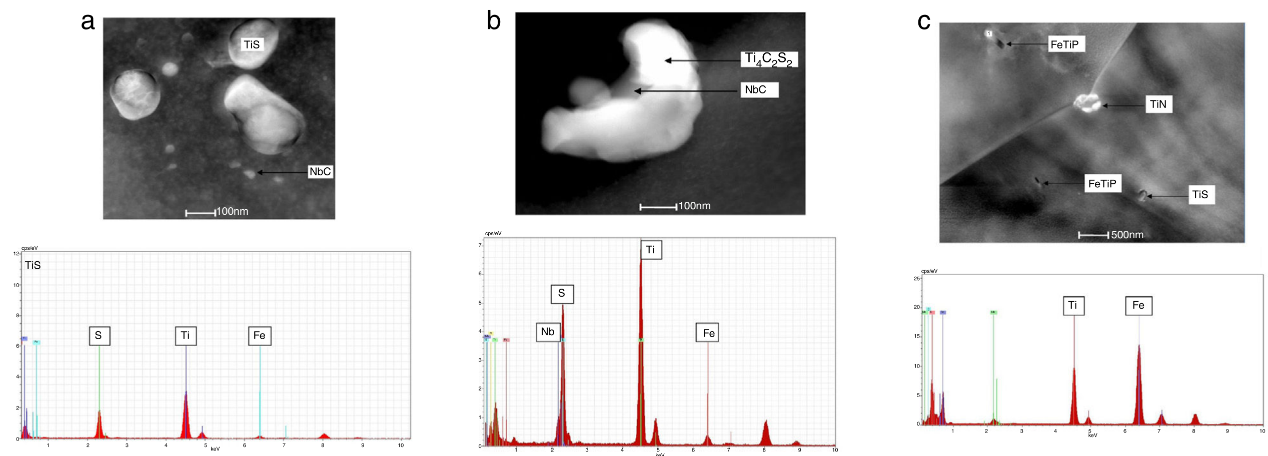

Fig. 3 – The main precipitates found in: (a) IF-Ti, (b) IF-TiNb, (c) IF-TiNbP.

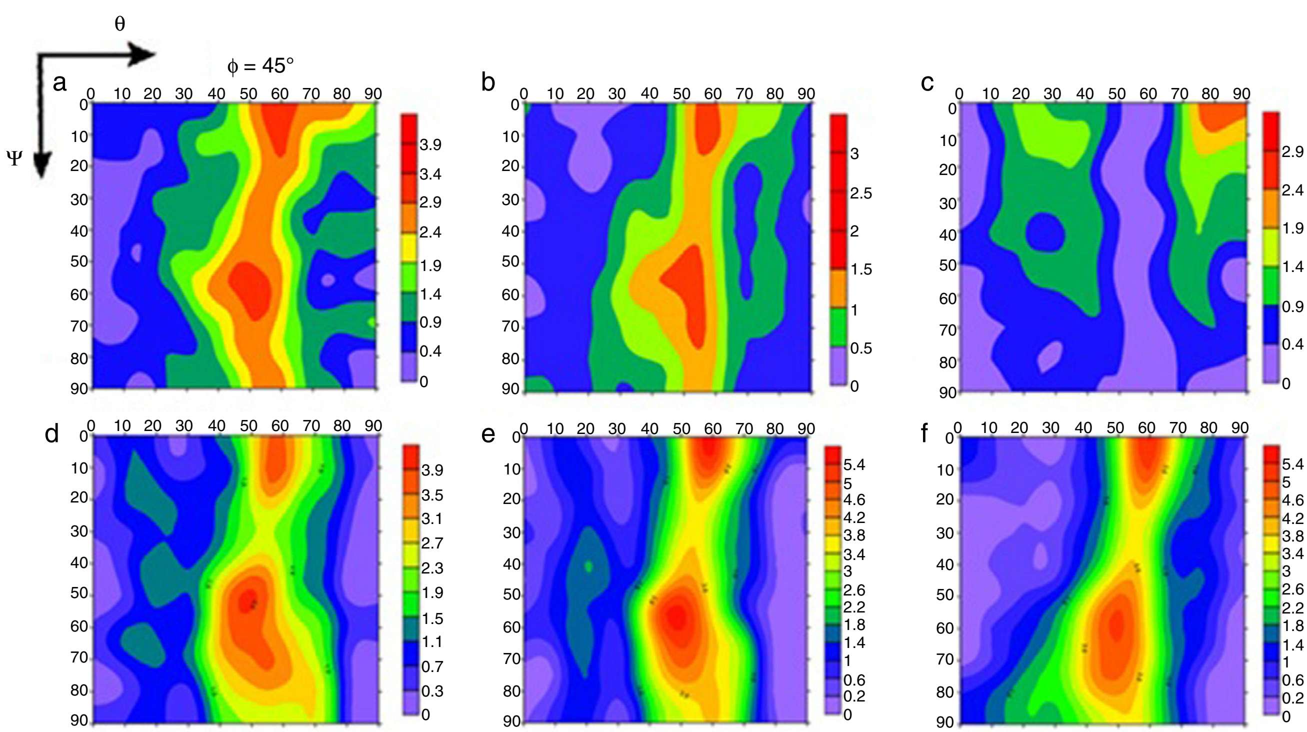

Fig. 4 – ODF's of the samples: measurements on the surface of (a) IF-Ti steel, (b) IF-TiNb steel, (c) IF-TiNbP steel; measurements in the half thickness, (d) IF-TiNb steel, (e) IF-TiNbP steel, (f) IF-TiNbP steel.

around 2.0 and planar anisotropy should be null, to assure the desired intensity of alpha and gamma fibers in IF steels, and to avoid discontinuities in thickness (normal anisotropy) or the formation of undesirable ears (planar anisotropy) in the final product.

IF steel with titanium additions presented a value of 1.92 for the normal anisotropy (Table 2). When analyzing the texture intensities in the ODFs from Fig. 4a and d, it can be seen that gamma fiber has the higher values of intensity (3.0 < intensity < 3.9) when compared to alpha fiber, both for surface and for half thickness measurements. Thus, the normal anisotropy seems to be in accordance with the texture developed after rolling. When analyzing the result for the planar anisotropy coefficient, however, it is verified that this one does not have a null value. This fact might be attributed to the presence of a Goss component, which is a shear component originated from non-homogeneous deformations. The Goss is defined by {011}⟨100⟩ orientation, which is considered harmful to the steel drawability. An ideal texture behavior for the deep drawing application includes an absence or low intensity of the Goss component in the material [10]. In fact, as presented by Daniel [11] and Lequeu [12], the presence of the Goss component is characterized by a strong tendency to contribute to high values of normal and planar anisotropy coefficients.

IF steel with additions of titanium and niobium exhibited different intensities for gamma fiber in the surface and in the half-thickness (Fig. 4b and e). Comparing the results obtained for planar and normal anisotropy to the IF-Ti steels, it was observed that the absolute values of these coefficients decreased. It is important to note that the gamma fiber from the surface of the rolled sheet of the IF-TiNb steel presents similar values throughout its length, being, therefore, more homogeneously distributed along the material. Gamma fiber improves formability and decreases the influence of the planar anisotropy coefficient on the material, mainly by the contribution of the {111}⟨112⟩ component, which in this case, presented a maximum value of 5.1 [13]. This might be the reason why the r_m value remained above 1.0 and the value of Δr decreased. Even so, the value of the planar coefficient remains high (in relation to the ideal value of zero) and, once again, that phenomenon can be associated with the presence of a Goss component.

IF steels with additions of titanium, niobium and phosphorus presented interesting results. The addition of phosphorus to IF steels aims to promote solid solution hardening to increase its resistance. However, there is a common concern that the presence of P in steels might lead to the occurrence of cracks and processing defects, but this is only true when specific amounts of P are added to steels with higher carbon content. Suzuki et al. [14] reported that phosphorus embrittlement only occurs in alloys with carbon contents higher that 0.25 wt% and for P contents higher than 0.03 wt%. This same effect was reported by Clyne et al. [15] during casting. Slower cooling rates during thermomechanical processing can prevent surface and inner cracks on these steels. Song [16] reported how processing should be carried to avoid such defects and also how the presence of Nb in the alloy can reduce the segregation of P at the grain boundary, diminishing the occurrence of cracks in the steel.

Analysis of the IF-TiNbP steel ODFs revealed that the rolling processes completely annulled the gamma fiber on the surface of the laminated sheet (Fig. 4c). However, this fiber was strongly favored in the half thickness (Fig. 4f). The sheet surface displayed the highest value found in all three steels, regarding the Goss component, reaching a level of 2.5. This result compromises the stamping properties, mainly because, as previously mentioned, gamma fiber has been completely suppressed in this same region. It is interesting to note that, despite the unsatisfactory result on the surface of the IF-TiNbP steel, the values of the fibers at half thickness are considered good in terms of deep drawing applications, i.e., low Goss value, high gamma fiber value and low alpha fiber value. The results obtained for normal and planar anisotropy coefficients presented the best combination of values among the three steels, despite the lower normal anisotropy value [17].

In fact, the IF-TiNbP steel presented the highest tensile strength, according to Table 2. Increased material strength can lead to increased friction between the sheet surface and the rolling rolls. This friction causes the generation of shear stresses, which leads to the nucleation of preferential orientations in the directions of the Goss component. This fact was observed in the works of Lee [18] and Bruna [19], who analyzed the effect of the interaction of cylinders with insufficient lubrication in the formation of shear components.

Phosphorus additions also cause the formation of FeTiP precipitates that consume the excess of titanium (which would form carbides), leaving carbon in solution within the matrix [20]. The presence of FeTiP precipitates was evidenced by STEM (Fig. 3c).

It is important to emphasize that the carbon in solution leads to the occurrence of discontinuities in the yield strength [21]. The combination of all those factors indicates that the presence of a strong Goss component on the surface is related to the observed discontinuous yield strength in IF-TiNbP steel (Fig. 2).

This interpretation is supported by the fact that a high amount of carbon in solution is associated with a decrease in the normal anisotropy coefficient due to the nucleation of unfavorable texture components, i.e., a decrease in the relative intensity of the gamma fiber [21]. In fact, Hölscher [22] observed that the elevation of carbon content in the matrix increases Goss orientation formation, while in the case of precipitated carbon, the Goss orientation density is strongly reduced. The reason for this fact is that the carbon in solution leads to more non-homogeneous deformation, resulting in shear bands.

5. Conclusions

Additions of phosphorus in the structure of an IF steel improved its resistance when compared to the other IF-Ti and IF-TiNb steels. IF-TiNbP steel presented better crystallographic texture results, concerning alpha and gamma fibers intensities measured at its half thickness. However, crystallographic texture analysis revealed clear differences between measurements taken from the surface and from the half thickness of the steel sheets. This fact indicates that special attention must be paid to lubrication during the rolling process, to avoid discrepancies along the thickness of the laminated sheet. The discontinuous yield strength in the IF-TiNbP steel suggests that carbon remained in solid solution, due to the formation of FeTiP precipitates. Therefore, in order to utilize the full potential for alloy strengthening and satisfactory crystallographic texture, resulted from the addition of phosphorus, IF steels must have their rolling process closely controlled to mitigate the effects of solid solution carbon and excessive hardening that might compromise their deep drawing applications.

Conflicts of interest

The authors declare no conflicts of interest.

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