Pearlite Transformation in a deformed TRIP/TWIP austenitic Steel

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Abstract

The increasing population of defects in strained austenite not only increases the heterogeneous nucleation site density for pearlite transformation, but also increases the austenite free energy due to the strain. This fact leads to a reduction in the critical free energy for pearlite nucleation, which triggers pearlite formation as compared to austenite transformation without straining. Besides, a significant refinement of the pearlite colony size due to hard impingement between neighboring colonies is achieved.

Keywords: Pearlite transformation; TRIP/TWIP steel; grain refinement; pearlite spherodization; medium manganese steel

Pearlite transformation has been widely studied in the last century and is one of the most well-known microstructures of steel [1-3]. According to the classical nucleation theory [4], nucleation of pearlite starts with a nucleus of ferrite or cementite in a grain

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boundary or an inclusion [5], and then a cementite or ferrite nucleus forms adjacent to the primary nucleus with a specific orientation relationship. Ferrite and cementite arrange themselves in a cooperative manner that results in alternate ferrite and carbide lamellae maintaining a crystallographic orientation relationship, which grow sidewise and edgewise to take over the austenite grain. Grain boundaries and inclusions act as nucleation sites due to the reduction of the energy necessary for nucleation by the destruction of the interface between these and the adjacent lattice. In the same way, other lattice defects such as twins or deformation bands contribute to the reduction of the energy for nucleation by the removal their interface [6]. However, just a few studies focus on these nucleation sites and their effect on pearlite transformation.

Furuhara et al [7] studied the effect of slight warm deformation of austenite prior to pearlite transformation, concluding that deformation effectively accelerates the intragranular nucleation of pearlite even if intragranular pearlite is hardly formed without deformation. Additionally, the authors observed that MnS particles activate as intragranular nucleation sites of pearlite.

Umemoto et al [8] studied the effect of warm rolling of austenite in pearlite transformation and concluded that deformation accelerates pearlite transformation. Besides the increase in the austenite grain surface area per unit volume by the elongation of grains, the authors suggested the possibility that twins or deformation bands generated during warm rolling act as potential nucleation sites for pearlite.

This last work was carried out in steels with chemical composition that hinder austenite at room temperature, and hence the effect of stain in austenite reported correspond to indirect determination techniques. By contrast, the work presented here is carried out in a TRIP/TWIP steel (chemical composition is listed in Table 1) where austenite is stable at room temperature. The steel studied here is related with the so-called lightweight

steels (Fe-Mn-Al-C system), which have received much attention by the scientific community given their potential for use in automotive sector [9-11]. In this context, the aim of this work is to deepen into the effect of austenite straining on pearlite transformation, in terms of kinetics and morphology, under different levels of prior austenite deformation.

The steel is casted and hence is hot-forged at 1050 °C to avoid the solidification microstructure. Hot forging at this temperature enhance the grain recrystallization promoting the formation of a very coarse austenitic grain of 250 μm in average diameter. The microstructure of the steel is fully austenitic at room temperature as indicated in the XRD spectrums shown in Fig. 1(a). XRD measurements were performed by means of a Bruker AXS D8 diffractometer equipped with a Co-K α X-ray tube with Goebel mirror optics and a LynxEye linear position sensitive detector for ultra-fast XRD measurements. Diffraction patterns were analyzed by Rietveld refinement using the TOPAS 4.2 software, where the crystallographic information of each phase was obtained from Pearson's crystal structure database for inorganic compounds.

Cylindrical samples of 5 mm in diameter and 4 mm in length were subjected to different levels of deformation in a 100 kN capacity electromechanical testing machine (Microtest EM2/FR) at a crosshead speed of 0.2 mm/min (polytetrafluoroethylene sheets of 75 μm in thickness are placed between the specimen and the plates to reduce friction and the related barreling effect). The levels of deformation achieved were 15, 35, 50 and 70 % reduction in-length. These were calculated as the ratio between the initial height and the crosshead displacement corrected to the compliance with standard for compression tests.

Figure 1: XRD spectrums of (a) As-received microstructure; (b) after 15% deformation, (c) after 35% deformation, and (d) after 70% deformation

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Figure 3. Deformation bands (black arrows) (a) and twins (white arrows) (b) promotes in the austenitic microstructure by deformation at 35%.

The morphology of the grain size also changes with deformation, lengthening perpendicularly to the direction of compression. Table 2 lists the quantitative results for each condition i.e., the aspect ratio of the austenite grains, the volume fractions of austenite and deformation induced martensite and the microstrain in austenite. Assuming a direct relation between lattice defects and microstrain in austenite, quantitative X-ray diffraction (XRD) analyses were used to determine the related microstrain. The spectrums collected are shown in Figure 1. As shown in Table 2, microstrain of austenite increases as deformation is raised. However, for deformation levels above the 35%, deformation induced martensitic transformation also occurs. The volume percent of martensite induced by deformation in each sample was quantified by XRD. Table 2 reveals that the higher the level of deformation, the higher the volume percent of martensite. For the lowest level of deformation (15%) no martensite was detected in the XRD diffractogram (see Figure 1(b)), although the microstructure shows local traces of martensite (see Figure 2(b)), whose presence is below the 3%, which is the XRD experimental error.

An isothermal heat treatment in the pearlitic field were carried out in a Carbolite CTF 12/65/550 furnace at 550 °C for 1 hour followed by quenching in water in a sample previously deformed at 35%. Figure 4 (a) shows the pearlitic microstructure obtained, revealing also the presence of martensite. The martensitic nature of the features observed in Fig 4(a) is consistent with the Electron backscatter diffraction (EBSD) analysis shown in Figure 4(b). EBSD maps were collected at an acceleration voltage of 20 kV and collected using a CRYSTAL detector of Oxford Instruments mounted in a SEM JEOL JSM6300 microscope. EBSD maps were generated in areas of about $346 \times 282 \mu\text{m}^2$ at step sizes of $0.45 \mu\text{m}$. The indexation of the Kikuchi lines and the determination of the orientations were done with the software CHANNEL 5 developed by HKL Technology. Figure 4(b) shows an IPF map where body-centered cubic (bcc) martensite is represented by red pixels, and face-centered cubic (fcc) austenite is represented by blue pixels. Additionally, Figure 4(c) shows the microstructure obtained after heat treatment of the area shown in Figure 4(a), consisting of pearlite (dark etching areas) tempered martensite (light etching regions) and a mixture of austenite and fresh martensite (white etching areas). Besides, Figure 4(d) shows details of the tempered martensite microstructure.

Figure 4. a) Austenitic and martensitic microstructure after deformation at 35%, b) EBSD Phase map. Red places represent BCC phase and blue places represent FCC phase. EBSD map was carry out in a samples deformed at 35% with an step size of 0.45 μm , c) same place of the microstructure a) after an isothermal treatment at 550°C for 1 hour in the pearlite field. d) SEM micrography of the tempered martensite obtained after the isothermal treatment.

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125 Figure 5 demonstrates the role that the different lattice defects present in austenite have
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Figure 5. Pearlite nucleated in a twin (a) and deformation bands (b)

Figure 6 shows optical micrographs of the samples transformed at 550 °C for 36 hours without prior deformation (Figure 6(a)) and deformed at 15%, 35%, 50% and 70% (Figure 6(b), 6(c), 6(d) and 6(e), respectively). The white etching areas correspond to austenite and/or fresh martensite, dark etching areas correspond to pearlite, and light etching areas correspond to tempered martensite.

It is easy to see that the area percent of pearlite increases with the increase in the degree of deformation. The area fraction of pearlite was evaluated on optical micrographs by point counting method according to ASTM E562-02. For the sample deformed at 35%, tempered martensite regions were distinguished of pearlite by metallographic procedures, and were therefore avoided in the evaluation of the pearlite area percent.

For high levels of deformation (50% and 70%) tempered martensite and pearlite areas are interlocked, which makes it difficult to distinguish pearlite and determine its amount by optical microscopy. Therefore, these two conditions were excluded from the study.

Figure 6. Optical micrographs of the sample transformed at 550°C for 36 hours without previous deformation (a) and the samples previously deformed at 15%, 35%, 50% and 70% (b, c, d and e respectively)

Figure 7(a) shows the total area percent of pearlite as a function of the previous deformation in austenite. Here, the higher the prior deformation of austenite is, the higher the volume fraction of pearlite obtained for the same isothermal heat treatment. The grain boundaries are, in all cases, covered by pearlite nodules. The evolution with time of the size of these nodules is similar in all the samples, indicating that kinetics of pearlite growth is not accelerated by prior austenite deformation. However, there is an increase in the fraction of intragranular pearlite with the increase in the degree of the deformation.

Figure 7. (a) Total area fraction of pearlite, and (b) area fraction of intragranular pearlite as a function of the deformation.

The fraction of intragranular pearlite was measured on optical micrographs using ImageJ software, excluding pearlite nucleated at grain boundaries and tempered martensite regions. Figure 7(b) shows the area fraction of intragranular pearlite as a function of deformation of the austenite. The size of intragranularly formed pearlite nodules presents a similar evolution with time as that of the size of pearlite nodules at grain boundaries. Nevertheless, the number density of intragranular pearlite nodules increases with the increase in the extent of prior austenite deformation.

The increase in the number of lattice defects, such as twins and deformation bands, increases the number of nucleation sites for pearlite nodules, and hence, a higher nucleation rate is obtained. In addition to increasing the density of heterogeneous nucleation sites, the strain energy of the defects raises the austenite free energy, which in turn leads to a specific reduction in the critical free energy for pearlite nucleation as it was reported by Maalekian et al [12]. As a result, pearlite starts to nucleate earlier in strained austenite as compared to pearlite nucleating in austenite without straining.

The effect of the deformation in the morphology of pearlite was observed in SEM micrographs. Figure 8 shows SEM micrographs of the samples transformed at 550 °C for 36 hours without deformation (Figure 8(a)) and previously deformed at 15%, 35%, 50% and 70% (Figures 8(b), 8(c), 8(d) and 8(e), respectively).

The surface of pearlite colony per unit volume of pearlite colonies ($S_V^{P/P}$) was measured by quantitative metallography, consisting in overlapping a circumference on SEM micrographs and counting the intersection with an edge of a colony (pearlite areas where the lamellae have the same direction [10]) using the following equation [11]:

$$S_V^{P/P} = \frac{2 \cdot n_r \cdot M}{\pi \cdot d_r} \quad (1)$$

Where d_r is the diameter of the circumference, n_r is the number of intersections with an edge of a colony and M is the magnification of the micrograph.

Figure 8. SEM micrographs of pearlite colonies of the samples previously deformed at: a) 0% b) 15% c) 32% d) 50% and e) 70%. Spheroidized pearlite is shown in (d) and (e).

Figure 9 shows the evolution of $S_V^{P/P}$ as a function of deformation of the austenite for the samples transformed at 550 °C for 36 hours. For high levels of deformation, the austenite microstructure has a higher amount of lattice defects, and a refinement of the colony size is observed. For samples deformed at 50% and 70%, pearlite does not show a laminar morphology, but a mixed morphology consisting in lamellar and spheroidal cementite as illustrated in Figure 8(d) for the case of the sample deformed at 50%, and completely spheroidized cementite for the case of the sample deformed at 70% (Figure 8(e)).

Figure 9. Area per unit volume of the pearlite colony as a function of the previous deformation

As aforementioned, the increase in $S_V^{P/P}$ suggests an increase in the number of the potential nucleation sites for pearlite. Once a pearlite colony is nucleated, pearlite lamellae grow until another colony growing in the opposite direction is found. At this point, the clamping of both occurs (hard impingement), and the growth of the colony stops. Therefore, the increase in the number of potential intragranular nucleation sites

by increasing the density of twins or deformation bands provides a colony size refinement because of the hard impingement events on growing colonies. For higher levels of deformation, such hard impingement events are so abundant that pearlite colonies barely grow. This results in a pearlitic microstructure formed by short lamellae of pearlite or just some nucleus of pearlite whose growth was so short that its geometric form is closer to that of a particle than to that of a lamellar structure, providing a microstructure of spheroidized morphology.

We might conclude that deformation of austenite prior to pearlite transformation effectively accelerates the intragranular nucleation of pearlite through the nucleation at twin boundaries and deformation bands. However, the increasing population of defects not only increases the density of heterogeneous nucleation sites for pearlite transformation, but also increases the austenite free energy due to strain. This fact leads to a reduction in the critical free energy for pearlite nucleation, which triggers pearlite formation. For the case of highly strained austenite, the refinement of the pearlite colony size is so high that a spheroidized morphology of pearlite is obtained.

Acknowledgments

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Figure Captions

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Tables

Table 1: Chemical composition (in wt%) of TRIP/TWIP steel studied

	C	Mn	Al	Fe
Wt. %	0.63	9.82	4.60	Balance

Table 2. Aspect ratio, volume fraction of martensite, volume fraction of austenite and microstrain austenite as a function of the deformation.

Deformation	0%	15%	35%	50%	70%
Aspect ratio	0.77 ± 0.09	0.67 ± 0.14	0.55 ± 0.15	0.31 ± 0.08	1.54 ± 0.42
Volume fraction Martensite	0 ± 3	0 ± 3	7.2 ± 3	27.6 ± 3	47.9 ± 3
Volume fraction austenite	100 ± 3	100 ± 3	92.8 ± 3	72.4 ± 3	51.5 ± 3
Microstrain austenite	0.035	0.096	0.12	0.14	0.24

(a)

(b)

(c)

(d)

(e)

200 μm

(a)

25 μm

(b)

50 μm

(a) 100 μm

(b) 20 μm

(c) 100 μm

(d) 1 μm

(a) 200 μm

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(d) 200 μm

(e) 100 μm

(a)

(b)

(c)

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(e)

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Figure 65 shows optical micrographs of the samples transformed at 550 °C for 36 hours without prior deformation (Figure 65(a)) and deformed at 15%, 35%, 50% and 70% (Figure 65(b), 65(c), 65(d) and 65(e), respectively). The white etching areas correspond to austenite and/or fresh martensite, dark etching areas correspond to pearlite, and light etching areas correspond to tempered martensite.

It is easy to see that the area percent of pearlite increases with the increase in the degree of deformation. The area fraction of pearlite was evaluated on optical micrographs by point counting method according to ASTM E562-02. For the sample deformed at 35%, tempered martensite regions were distinguished of pearlite by metallographic procedures, and were therefore avoided in the evaluation of the pearlite area percent. For high levels of deformation (50% and 70%) tempered martensite and pearlite areas are interlocked, which makes it difficult to distinguish pearlite and determine its amount by optical microscopy. Therefore, these two conditions were excluded from the study.

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Figure 65. Optical micrographs of the sample transformed at 550°C for 36 hours without previous deformation (a) and the samples previously deformed at 15%, 35%, 50% and 70% (b, c, d and e respectively)

Figure 76(a) shows the total area percent of pearlite as a function of the previous deformation in austenite. Here, the higher the prior deformation of austenite is, the higher the volume fraction of pearlite obtained for the same isothermal heat treatment. The grain boundaries are, in all cases, covered by pearlite nodules. The evolution with time of the size of these nodules is similar in all the samples, indicating that kinetics of pearlite growth is not accelerated by prior austenite deformation. However, there is an increase in the fraction of intragranular pearlite with the increase in the degree of the deformation.

Figure 76. (a) Total area fraction of pearlite, and (b) area fraction of intragranular pearlite as a function of the deformation.

The fraction of intragranular pearlite was measured on optical micrographs using ImageJ software, excluding pearlite nucleated at grain boundaries and tempered martensite regions. Figure 76(b) shows the area fraction of intragranular pearlite as a function of deformation of the austenite. The size of intragranularly formed pearlite nodules presents a similar evolution with time as that of the size of pearlite nodules at grain boundaries. Nevertheless, the number density of intragranular pearlite nodules increases with the increase in the extent of prior austenite deformation. The increase in the number of lattice defects, such as twins and deformation bands, increases the number of nucleation sites for pearlite nodules, and hence, a higher nucleation rate is obtained. In addition to increasing the density of heterogeneous nucleation sites, the strain energy of the defects raises the austenite free energy, which in turn leads to a specific reduction in the critical free energy for pearlite nucleation as it was reported by Maalekian et al [129]. As a result, pearlite starts to nucleate earlier in strained austenite as compared to pearlite nucleating in austenite without straining.

The effect of the deformation in the morphology of pearlite was observed in SEM micrographs. Figure 87 shows SEM micrographs of the samples transformed at 550 °C for 36 hours without deformation (Figure 87(a)) and previously deformed at 15%, 35%, 50% and 70% (Figures 87(b), 87(c), 87(d) and 87(e), respectively). The surface of pearlite colony per unit volume of pearlite colonies ($S_V^{P/P}$) was measured by quantitative metallography, consisting in overlapping a circumference on SEM micrographs and counting the intersection with an edge of a colony (pearlite areas where the lamellae have the same direction [10]) using the following equation [11]:

$$S_V^{P/P} = \frac{2 \cdot n_r \cdot M}{\pi \cdot d_r} \quad (1)$$

Where d_r is the diameter of the circumference, n_r is the number of intersections with an edge of a colony and M is the magnification of the micrograph.

Figure 87. SEM micrographs of pearlite colonies of the samples previously deformed at a) 0% b) 15% c) 32% d) 50% and e) 70%. Spheroidized pearlite is shown in (d) and (e).

Figure 98 shows the evolution of $S_V^{P/P}$ as a function of deformation of the austenite for the samples transformed at 550 °C for 36 hours. For high levels of deformation, the austenite microstructure has a higher amount of lattice defects, and a refinement of the colony size is observed. For samples deformed at 50% and 70%, pearlite does not show a laminar morphology, but a mixed morphology consisting in lamellar and spheroidal cementite as illustrated in Figure 87(d) for the case of the sample deformed at 50%, and completely spheroidized cementite for the case of the sample deformed at 70% (Figure 87(e)).

Figure 98. Area per unit volume of the pearlite colony as a function of the previous deformation

As aforementioned, the increase in $S_V^{P/P}$ suggests an increase in the number of the potential nucleation sites for pearlite. Once a pearlite colony is nucleated, pearlite lamellae grow until another colony growing in the opposite direction is found. At this point, the clamping of both occurs (hard impingement), and the growth of the colony stops. Therefore, the increase in the number of potential intragranular nucleation sites

by increasing the density of twins or deformation bands provides a colony size refinement because of the hard impingement events on growing colonies. For higher levels of deformation, such hard impingement events are so abundant that pearlite colonies barely grow. This results in a pearlitic microstructure formed by short lamellae of pearlite or just some nucleus of pearlite whose growth was so short that its geometric form is closer to that of a particle than to that of a lamellar structure, providing a microstructure of spheroidized morphology.

We might conclude that deformation of austenite prior to pearlite transformation effectively accelerates the intragranular nucleation of pearlite through the nucleation at twin boundaries and deformation bands. However, the increasing population of defects not only increases the density of heterogeneous nucleation sites for pearlite transformation, but also increases the austenite free energy due to strain. This fact leads to a reduction in the critical free energy for pearlite nucleation, which triggers pearlite formation ~~as compared to austenite to pearlite transformation without straining~~. For the case of highly strained austenite, the refinement of the pearlite colony size is so high that a spheroidized morphology of pearlite is obtained.

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Figure Captions

Figure 1: XRD spectrums of (a) As-received microstructure; (b) after 15% deformation, (c) after 35% deformation, and (d) after 70% deformation

Figure 24. Initial microstructure (a) and microstructure obtained after deformation at 15% (b), 35% (c), 50% (d) and 70% (e).

Figure 32. Deformation bands (black arrows) (a) and twins (white arrows) (b) promotes in the austenitic microstructure by deformation at 35%.

Figure 43. a) Austenitic and martensitic microstructure after deformation at 35%, b) EBSD Phase map. Red places represent BCC phase and blue places represent FCC phase. EBSD map was carry out in a samples deformed at 35% with an step size of 0.45 μm , c) same place of the microstructure a) after an isothermal treatment at 550°C for 1 hour in the pearlite field. d) SEM micrography of the tempered martensite obtained after the isothermal treatment.

Figure 54. Pearlite nucleated in a twin (a) and deformation bands (b)

Figure 65. Optical micrographs of the sample transformed at 550°C for 36 hours without previous deformation (a) and the samples previously deformed at 15%, 35%, 50% and 70% (b, c, d and e respectively)

Figure 76. (a) Total area fraction of pearlite, and (b) area fraction of intragranular pearlite as a function of the deformation.

Figure 87. SEM micrograhs of pearlite colonies of the samples previously deformed at. a) 0% b) 15% c) 32% d) 50% and e) 70%. Spherodized pearlite is shown in (d) and (e).

Figure 98. Area per unit volume of the pearlite colony as a function of the previous deformation

Tables

Table 1: Chemical composition (in wt%) of TRIP/TWIP steel studied

	C	Mn	Al	Fe
Wt.%	0.63	9.82	4.60	Balance

Table 2. Aspect ratio, volume fraction of martensite, volume fraction of austenite and microstrain austenite as a function of the deformation.

Deformation	0%	15%	35%	50%	70%
Aspect ratio	0.77 ± 0.09	0.67 ± 0.14	0.55 ± 0.15	0.31 ± 0.08	1.54 ± 0.42
Volume fraction Martensite	0 ± 3	0 ± 3	7.2 ± 3	27.6 ± 3	47.9 ± 3
Volume fraction austenite	100 ± 3	100 ± 3	92.8 ± 3	72.4 ± 3	51.5 ± 3
Microstrain austenite	0.035	0.096	0.12	0.14	0.24