Stress or Strain Induced Martensitic and Bainitic transformations during ausforming processes

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Abstract: The so-called ausforming treatment consists in plastically deforming a fully austenitized steel below the recrystallization stop temperature, prior to either a martensitic or a bainitic transformation. Although this procedure is envisioned as a way to improve the mechanical response of the attained microstructure, it is not without its drawbacks, as the possibility of phase transformations occurring during the deformation step. When such step is applied at relatively higher temperatures than those of the aimed microstructure, the presence of those softer phases could be rather detrimental for the final microstructure. The current study aims to further study those phase transformations to analyze whether they are induced via either stress or strain and to identify the temperature ranges in which they tend to occur, so that they can be avoided or used to improve the final microstructure properties in the future. A final evaluation on the impact of these induced transformations on the final microstructure when deformation is followed by either cooling or an isothermal treatment is also performed.

Keywords: ausforming; thermomechanical treatments; compression; dilatometry; strain induced transformations; stress induced transformations

1. Introduction

Ausforming processes are those thermomechanical treatments in which austenite (γ) is plastically deformed before either a martensitic (α') or a bainitic (α_B) transformation takes place [1-4]. Although they have many benefits, such as the refinement of microstructures or the acceleration of the corresponding phase transformation [4-7], there is a possibility of stress or strain induced transformations during the deformation step that could alter the final mechanical properties.

As the application of deformation can alter the temperatures at which transformations start, martensite, bainite or even ferrite can form at temperatures at which they usually do not appear [8-16]. Although stress and strain induced martensitic transformations were discovered almost one century ago by Scheil [17], they have been mainly studied in alloys in which austenite is stable at room temperature until stresses or strains are applied to it, see for example the works performed on iron-nickel alloys or austenitic steels in references [17-27]. In 1967, Zackay et al. [28] showed that these stress or strain induced transformation phenomena were also happening in high strength steels formed by other phases than austenite. The authors showed that this phenomenon, called Transformation Induced Plasticity (TRIP), enhances the ductility of those steels by additional strain hardening due to the transformation $\gamma \rightarrow \alpha'$. Later on, Sugimoto et al. [29] also showed that it was not only martensite which could form under straining, but also bainite, as they detected its formation when performing high temperature

tensile tests to dual phase steels. Their research on TRIP effect has been verified by many others [30-39].

However, there are few authors who have devoted their work to study these transformations from a 100% austenitic phase which is strained at such high temperatures as the ones at which ausforming processes are applied, and even less to study these transformations in austenitized steels. For instance, although the bainitic transformation has been proved to be promoted under elastic [40, 41] or plastic strains [42], the outcome of such deformation is still uncertain and cannot be predicted yet.

There are, though, some examples in the literature where ausforming treatments show deviations in the stress-strain curves, besides some unexpected trends in terms of volume fractions or hardness values [7, 43]. He et al. [7] carried out ausforming treatments, in which austenite was deformed up to different strains before isothermally treating the steel. For the highest deformation temperature, i.e. 700 °C, temperature for which the highest strains were applied, i.e. about 45 %, a deviation can be seen in the last stages of the stress-strain curve. The amount of α_B was determined by quantitative metallography and by dilatometry and the only condition where a disagreement between both measurements was found was for the mentioned condition in which austenite was deformed up to 45 % at 700 °C. The authors could not explain why the results did not match, which suggests that the reason lying beneath may be related to the presence of stress or strain induced bainite formed during the deformation step.

We also found similar occurrences elsewhere [43]. In that work, three ausforming temperatures (T_{def}) were selected, i.e. 520 °C, 400 °C and 300 °C,

where the first one was found to be in the hiatus between the ferrite/pearlite region and the bainitic region, whereas the last two temperatures were within the bainitic region, i.e. between M_s and B_s temperatures, martensite and bainite start temperatures respectively. To study the austenite hot deformation behavior, samples were austenitized and then cooled down to the corresponding T_{def} , which was held for 15 s to allow the system and sample to stabilize. After that holding, samples were compressed up to the highest attainable strain of the equipment and then cooled down to room temperature, see sketch D+C in Figure 1(a). When analyzing the stress-strain curves, deviations from the curve were observed, see Figure 1(b), which was attributed to the formation of bainite during the deformation process, although no deeper study was performed at that time. In order to minimize such possibility, a rather low strain was selected, 10%, and, even then, microstructural observations and some unexpected hardness values indicated that there could have been phase transformations taking place during the deformation step that affected the microstructure, see Ref. [43].

However, it is still not clear how much the presence of those unexpected phases can alter the final microstructure properties which the thermomechanical treatment was originally designed for. In the current work, a thorough study is performed on phase transformations taking place during single hit compression tests at 520, 400 and 300 °C (same temperatures as in Ref. [43]). The study involves a thermodynamics-based rationalization and an experimental observation, as well as a discussion about the reasons lying beneath the occurrence of this type of transformations. A final evaluation on the impact of these induced transformations on the final microstructure when either cooling or

isothermal treatments are performed is also carried out. Anticipating such phase transformations could be extremely useful when designing thermomechanical treatments as it would allow to either avoid them or to use them to improve the behavior and the final microstructure.

Figure 1. (a) Sketch of the thermal and thermomechanical treatments performed for this study, where C, D+C, I and D+I stand for Cooling, Deformation + Cooling, Isothermal, Deformation + Isothermal; (b) Stress-strain curves obtained during single hit compression test at $T_{\text{def}} = 520$, 400 and 300 °C. Adapted figure from results in Ref. [43].

2. Materials and Methods

For this study, a Sidenor's commercial alloy, SCM40, with chemical composition Fe-0.4C-3Si-1Cr-0.9(Mn+Mo), all in wt. %, has been selected.

Both thermal and thermomechanical tests have been performed in a high-resolution dilatometer Bahr 805D, using specimens 10 mm long and 4 and 5 mm in diameter, respectively. The dilatometer allows to analyze phase transformations by monitoring the change in length of the sample with time and temperature. The temperature is measured by type K thermocouples which are

welded to the central part of the sample and it can be controlled by two different ways: whereas an induction heating coil allows to increase the temperature, this can be decreased by blowing Helium to the sample, enabling to control the temperature and to apply the selected heating/cooling rates. The changes in length are measured by silica push-rods in direct contact with the sample. When samples must be subjected to compression, a specific module equipped with silicon nitride punchers has to be used. A thin film of molybdenum is usually placed in between the punchers and the sample to reduce the friction and the thermal gradient. Among other benefits, the dilatometric data allows to determine some critical temperatures. In this work, the M_s has been determined by the offset method explained in Ref. [44], which consists in defining the M_s temperature as the temperature at which the temperature-time curve deviates by a certain value, i.e. 0.2 % in our particular case, from the linearity.

X-Ray diffraction analyses were performed in selected bainitic microstructures obtained after performing some of the treatments, allowing to identify and to measure the phases volume fractions. To that end, a X-Ray Diffractometer Bruker AXS D8, which uses Co radiation, has been used. Its intensity and voltage were 30mA and 40kV, respectively, and measurements were taken in a range $2\theta=35-135^\circ$. To quantify the different phases volume fractions, all the areas under the peaks, i.e. (002), (112) and (022) for ferrite, and (200), (220) y (311) for austenite, were considered.

A Scanning Electron Microscope FEG-SEM JEOL JSM-6500, operating at 10kV, was used to study the microstructure in the central part of the sample transverse and (in some cases) the longitudinal sections, where the local plastic

deformation is maximum [45], since the local deformation does not equal the macroscopic one along the sample due to the barreling effect.

Samples for FEG-SEM observation were metallographically prepared by standard methods, followed by cycles of etching and polishing for better definition of the microstructural features that can be distorted during the grinding step. Finally, they were etched by using a 2% nital solution.

The procedure explained in Ref. [46] was used to measure the plate thickness of some of the bainitic microstructures on both the transverse and the longitudinal sections. It consists in estimating the mean linear intercept (MLT) by making measurements on SEM micrographs and subsequently applying a stereographic correction to MLT.

Vickers hardness measurements were made, after applying a load of 10 kg, and the reported value corresponds to the mean of at least 3 values, whereas its 95 % confidence interval.

The software MTDATA, which relies on the NPL-plus database for steels [47], enabled to perform all thermodynamic calculations.

3. Theory and calculations

As widely known, in the absence of stress or strain, the martensitic and bainitic transformations occur in temperature ranges which are defined by M_s , above which martensite can never form, and by B_s , which is the upper limit for bainitic transformation. However, the application of stress and deformation (either plastic or elastic) can shift such ranges to higher temperatures [40, 48-55].

In order to study all the possible scenarios, two different types of transformations that can occur during deformation have to be distinguished:

stress and strain induced transformations. The stress induced transformations are those that occur under the application of a load which always lies below the corresponding bulk austenite yield strength, whereas martensite/bainite is strain induced if dislocation slip in austenite precedes their formation [56]. Note that this classification only focuses on the stage that the transformation has taken place at, which simplifies the analysis, although it does not describe the mechanisms that have triggered the corresponding phase transformations, as does the classification stated by Chatterjee and Bhadeshia [57].

Therefore, several important temperatures must be defined: the B_d and M_d temperatures, which are the maximum temperatures above which no bainitic/martensitic transformation can occur, respectively, regardless the deformation that is applied [12-14]; and the $M_s(\sigma)$ and $B_s(\sigma)$, which are the temperatures above which the martensitic/bainitic transformations occur at stresses higher than the austenite yield point, σ_Y [58]. Both $M_s(\sigma)$ and $B_s(\sigma)$ change with the type of deformation, i.e. it is higher in pure shear than in uniaxial tension [59].

Therefore, in consonance with the classification that has been used in this work, there are several regions of interest depending on the stress level and the temperature that the phase transformation occurs at, see Figure 2. If such stress is below σ_Y , and it is applied in between M_s and $M_s(\sigma)$, then a stress induced martensitic transformation happens. For $T > M_s(\sigma)$, temperatures still in the bainitic range, large constant stresses below σ_Y kept for prolonged times have proved to accelerate the bainitic transformation and to promote the formation of small volume fractions of bainitic ferrite at temperatures where otherwise bainitic

ferrite was never attained in the absence of stress, temperatures in the range B_s - $B_s(\sigma)$ [40, 41, 60]. Although the application of stress can promote the bainitic transformation, it still requires time for the bainitic ferrite to nucleate and grow. For that reason, no bainite formed in the elastic region during a tensile/compression test at high temperature has been found, as the elastic range is usually overcome after few seconds. If stresses above σ_Y are applied at temperatures below M_d and above M_s , then strain induced martensitic transformations occur, whereas strain induced bainitic transformations become predominant for higher temperatures up to B_d . The mentioned critical temperatures have been calculated based on thermodynamics in the following subsections.

Figure 2. Schematic representation of the different phase transformations that can take place under the application of constant/increasing stress, as a function of the deformation temperature and the stress which the phase transformation starts at. σ_Y stands for yield strength. *Obtained under continuous deformation or under constant load. **Obtained only if temperature and stress are held constant for long times.

3.1. Calculation of non-deformation-related critical temperatures

If neither stresses nor strains are applied, the martensitic transformation occurs when temperature reaches the M_s , as previously mentioned. The energy condition that is satisfied below M_s is [61]:

$$\Delta G^{\gamma \rightarrow \alpha} \leq \Delta G_N^{\alpha'} \quad (1)$$

where $\Delta G^{\gamma \rightarrow \alpha}$ is the total driving force of the system (which equals the chemical driving force, $\Delta G_{ch}^{\gamma \rightarrow \alpha}$, for the stress-free strain-free state) and $\Delta G_N^{\alpha'}$ is the critical driving force needed to stimulate martensite by an athermal, diffusionless nucleation and growth mechanism, i.e. it equals the driving force for the transformation from austenite to ferrite at the M_s temperature. While $\Delta G_N^{\alpha'}$ has been assumed to be constant for low alloy steels [62, 63], the following formula derived by Ghosh and Olson has been widely used for steels containing larger solute concentrations [61]:

$$\begin{aligned} \Delta G_N^{\alpha'} = & \\ & = -1010 - \sqrt{(4009X_C^{1/2})^2 + (3097X_N^{1/2})^2} \\ & - \sqrt{(1868X_{Cr}^{1/2})^2 + (1980X_{Mn}^{1/2})^2 + (1418X_{Mo}^{1/2})^2 + (1653X_{Nb}^{1/2})^2 + (1879X_{Si}^{1/2})^2} \\ & \quad + (1473X_{Ti}^{1/2})^2 + (1618X_V^{1/2})^2 - \sqrt{(280X_{Al}^{1/2})^2 + (752X_{Cu}^{1/2})^2} \\ & \quad + (172X_{Ni}^{1/2})^2 + (714X_W^{1/2})^2 + 352X_{Co}^{1/2} \end{aligned} \quad (2)$$

where X_i is the mole fraction of the element i . On the contrary, the transformation to bainite follows different conditions, as the time that it takes for the nucleus to

form and grow may be long, i.e. even hours, and as its nucleation happens under paraequilibrium conditions. Therefore, the conditions that govern the bainitic nucleation and growth, respectively are [1]:

$$\Delta G^{\gamma \rightarrow \gamma' + \alpha} \leq \Delta G_N \quad (3)$$

$$\Delta G^{\gamma \rightarrow \alpha} \leq G_{SB} \quad (4)$$

where $\Delta G^{\gamma \rightarrow \gamma' + \alpha}$ is the driving force for the nucleation under paraequilibrium conditions. In addition, in this case, the critical driving force for the nucleation, ΔG_N , is a function of the temperature, as follows [64]:

$$\Delta G_N = 3.5463T_h - 3499.4 \text{ [J mol}^{-1}\text{]} \quad (5)$$

where T_h is the temperature in Kelvin. Regarding the value ΔG_{SB} , it is the stored energy of bainite (in the range 200-600 J mol⁻¹ [1, 65, 66]). The variability of ΔG_{SB} has been considered to calculate all critical temperatures related to bainite transformation, giving an average value followed by an uncertainty which equals its standard deviation. The highest temperature at which both conditions are satisfied corresponds to the Bs. Therefore, the Ms temperature is defined as the intersection of $\Delta G^{\gamma \rightarrow \alpha}$ and $\Delta G_N^{\alpha'}$, whereas the Bs is the lowest temperature at which Equations (3) and (4) are satisfied.

3.2. Stress-induced transformations

To establish the temperatures ranges in which stress induced transformations occur, the same energy balances found in Equations (1), (3) and (4) can be used. As it has been previously assumed that applied stress only interacts with the growth process [67], Equation (3) can be applied directly,

whereas small modifications must be made in Equations (1) and (4). In this case, the total driving force of the system is not only composed of $\Delta G_{ch}^{\gamma \rightarrow \alpha}$, but also of the mechanical energy, ΔG_{mech} , assisting the transformation:

$$\Delta G^{\gamma \rightarrow \alpha} = \Delta G_{ch}^{\gamma \rightarrow \alpha} - \Delta G_{mech} \quad (6)$$

This mechanical driving force introduced by the compressive stress was defined by Patel and Cohen [68] and it depends on the angle θ that the normal to the potential habit plane forms with the compressive stress direction, as follows:

$$\Delta G_{mech} = \frac{1}{2} s \sigma \sin 2\theta - \frac{1}{2} \zeta \sigma (1 + \cos 2\theta) \quad (7)$$

where s and ζ are the shear and dilatational strains associated with the martensitic or bainitic transformation and σ is the stress that is being applied in Pa. Different values for s and ζ for bainite, nanobainite and martensite have been reported in the literature; see *Table 1*. To consider this variability, calculations have been made by assuming the ranges $\zeta=0.03-0.04$ and $s=0.20-0.24$ for martensite and $\zeta=0.03$ and $s=0.22-0.26$ for bainite. Results have been averaged, being the calculation uncertainty the standard deviation of the calculation. Note that the values of s and ζ reported for nanobainite have not been considered as nanobainite is only obtained at low temperatures (just above M_s) [69] and, therefore, far from the temperature range used in this work. As ΔG_{mech} depends on the angle of the corresponding potential habit and we are concerned with the plates that form first, we have taken the maximum values of ΔG_{mech} for our calculations, which can be calculated by replacing θ by $\arctan\left(\frac{s}{\zeta}\right)/2$ [21]. The values of θ for a maximum ΔG_{mech} are included in *Table 1*.

The result must be converted from Pa to J mol⁻¹ by multiplying it by the molar volume at each temperature, i.e. $V_m = \frac{N \cdot a_{fcc}}{n_{fcc}}$, where N is Avogadro's number, n_{fcc} is the number of atoms per fcc unit cell, i.e. 4, and a_{fcc} is the austenite lattice parameter at the given temperature. Such lattice parameter has been calculated by using the austenite thermal expansion coefficient ($\alpha = 2.07 \cdot 10^{-5} \text{ }^\circ\text{C}^{-1}$) reported by Podder et al. [70] to extrapolate the room temperature lattice parameter, $a_{fcc}^{T_{room}}$, calculated by the expression developed by Dyson and Holmes [71], to the temperature of interest by the expression: $a_{fcc} = a_{fcc}^{T_{room}}(1 + \alpha(T - T_{room}))$, where T_{room} is the room temperature ($T_{room} = 25 \text{ }^\circ\text{C}$) and T is in $^\circ\text{C}$.

Table 1. Shape deformations s and ζ reported by different authors, depending on the product phase.

Transformation	s	ζ	θ for maximum $\Delta G_{mech} / ^\circ$	Reference
Bainite	0.22	0.03	41.12	[72]
	0.26	0.03	41.71	[73]
	0.24	0.03	41.44	[74]
Nanobainite	0.45	0.03	43.09	[75]
Martensite	0.20	0.04	39.35	[68]
	0.24	0.03	41.44	[76]

The stress σ must be set as the austenite yield strength at any given temperature to consider the limit scenario for martensite or bainite stress induced transformations. The experimental values cannot be used to find an expression that describes the σ_Y evolution with temperature because, as stress-strain curves representative of stress induced transformations are characterized by a

transition from parabolic to sigmoidal as transformations progresses [8-11], such transformations would lower σ_Y and would make the calculation invalid. For that reason, the latest developed model [77] has been used to predict σ_Y as a function of deformation temperature T_{def} and strain rate $\dot{\epsilon}$:

$$\begin{aligned} \sigma_Y = & (52.3 + 47.0X_C + 31.3X_{Si} + 1.0X_{Mn} + 1.0X_{Cr} + 31.3X_{Mo} + 1.3X_{Ni} \\ & + 10.7X_V + 3.8X_{Al} + 16.8X_{Cu} + 15.2X_{Ti} \\ & + 53(\pm 34)) \left(1 + 0.001 \ln \left(\frac{\dot{\epsilon}}{0.001} \right) \right) \left(1 - \left(\frac{T_{def} - 25}{1487 - 25} \right)^{0.658} \right) \end{aligned} \quad (8)$$

where X_i is the solute content of the element i in at. %. This model has been proved to be more accurate than other previous models for the prediction of the austenite yield strength in a wide compositional range, including the chemical composition of the steel used in this work [77]. As detailed in the same reference, the reported uncertainty of σ_Y includes its variability with the prior austenite grain size. This uncertainty has also been taken into account when calculating the uncertainty of both $M_s(\sigma)$ and $B_s(\sigma)$.

3.3. Strain-induced transformations

Finally, the M_d and B_d temperatures have been calculated. Although using the theoretical approach derived by Sherif et al. [78] to calculate M_d was explored, it was discarded as a reliable method because it became clear that only applies for microstructures in which austenite is a second phase. For that reason, a second approach has been adopted for the calculation of the M_d temperature. This approach, which has also been applied to calculate the B_d temperature, consists in applying the same energy balances that have been applied in previous subsections.

As happened with the stress-induced transformations, the stress that the austenite undergoes promotes the martensitic/bainitic transformations as it decreases the system energy by ΔG_{mech} . However, this time the austenite is also plastically deformed because stresses higher than austenite σ_Y are applied. Although the nucleation of bainite is promoted by the presence of a larger amount of defects or slip bands [79] which are generated during plastic deformation and act as nucleation sites [1], it has not been reported how the nucleation condition found in Equation (3) is modified because of the increase of nucleation sites. For that reason, the bainitic nucleation has not been assessed in this section, meaning that any calculation at temperatures higher than the one at which $\Delta G^{\gamma \rightarrow \gamma' + \alpha} = \Delta G_N$ is not valid.

Regarding growth, the dislocations introduced during the deformation process slow the progress of the glissile interfaces that must move in order for the martensite/bainite to grow by the so-called mechanical stabilization phenomenon. In this case $\Delta G^{\gamma \rightarrow \alpha}$ is the same that was defined in the last subsection, see Equation (6), although the growth balances described in Equations (1) and (4) are modified:

$$\Delta G^{\gamma \rightarrow \alpha} \leq \Delta G_N^{\alpha'} - \Delta G_{disl} \quad (9)$$

$$\Delta G^{\gamma \rightarrow \alpha} \leq \Delta G_{SB} - \Delta G_{disl} \quad (10)$$

Where ΔG_{disl} is the additional driving force needed so that the interface overcomes the dislocation density introduced by the plastic deformation [80-82]:

$$\Delta G_{disl} = \frac{\mu_T b_T (\rho^{1/2} - \rho_0^{1/2})}{8 \pi (1 - \nu)} \quad (11)$$

In this equation, ν is the Poisson ratio, taken as 0.27, ρ_0 and ρ are the dislocation densities for the initial moment, i.e. the yield point, and for any given strain, respectively, and b_T and μ_T are the Burgers vector and the shear modulus of austenite at the corresponding temperature T , given by:

$$b_T = b_0(1 + \alpha T) \quad (12)$$

$$\mu_T = \mu_0 \left[1 + \left\{ \frac{T - 300}{T_m} \right\} \left(\frac{T_m}{\mu_0} \right) \left(\frac{d\mu_T}{dT} \right) \right] \quad (13)$$

The values of b_0 and μ_0 are $2.5 \cdot 10^{-10}$ m and $8.1 \cdot 10^{10}$ J m⁻³, respectively, $\left(\frac{T_m}{\mu_0} \right) \left(\frac{d\mu_T}{dT} \right) = -0.91$ [83] and α and T_m are the thermal expansion coefficient and the austenite melting temperature. All the temperatures must be in Kelvin. The dislocation density in m⁻² at any given plastic strain ϵ_p can be calculated by the expression proposed by Maalekian et al. [80]:

$$\rho = \rho_0 + 2 \cdot 10^{14} \epsilon_p \quad (14)$$

where $\rho_0 = 2 \cdot 10^{13}$ m⁻².

To obtain the evolution of stress σ with plastic strain at any temperature and calculate ΔG_{mech} with it, a model must be used. In this work, the Johnson and Cook (JC) [84] model has been used. The JC is a phenomenological constitutive model which allows to predict the stress-strain curve at a given temperature T and under a fixed strain rate $\dot{\epsilon}_p$. It requires a low amount of material constants and, although it was initially proposed as a model to predict the flow stress behavior for metals subjected to large strains, high strain rates and high temperatures, it has successfully been applied for various materials, including steel [85], for different ranges of temperature and strain rates [86-88]. The JC model reads as follows:

$$\sigma = (A + B \varepsilon_p^n) \cdot \left(1 + C \ln \left(\frac{\dot{\varepsilon}_p}{\dot{\varepsilon}_{p0}} \right) \right) \cdot \left(1 - \left(\frac{T - T_r}{T_m - T_r} \right)^m \right) \quad (15)$$

where σ is the stress, $\dot{\varepsilon}_{p0}$ is the reference strain rate, T_r and T_m are the room and the melting temperatures and A , B , C , n and m are the model parameters, where A is the yield strength for $T = T_r$ and $\dot{\varepsilon}_p = \dot{\varepsilon}_{p0}$. The parameters A and m must be fitted from values of σ_Y at different temperatures. The value of m indicates the dependence of the yield strength with temperature, where $m=1$ means linear dependency. With those fitted values A and m , the plastic part of the curve can be fitted as well. The data used for the fittings must not have been altered by any stress or strain induced transformation, which means that only experimental σ_Y values obtained at temperatures lying between $M_s(\sigma)$ and B_s or higher than $B_s(\sigma)$ can be used and that any only stress strain curves which do not present deviations are valid. In case that only stress strain curves with deviations have been collected, the curve corresponding to the highest temperature up to the inflection point can be fitted.

The critical temperatures M_d and B_d are by definition such that no transformation occurs under plastic deformation (regardless the deformation level). However, due to the equipment limitations, it is only possible to affirm that the JC model is valid over the strain range that has been tested (up to the maximum strain attainable by the equipment) and it cannot be anticipated how the material would behave for higher strain levels. For that reason, and similarly to the well-established criteria of the M_d30 temperature, which has been defined as the temperature at which 50 % of martensite is formed after a true strain of 30 % [89-91], two new critical temperatures, M_d^* and B_d^* , have been defined as the

temperatures above which strain induced martensitic or bainitic transformations are not possible for a certain true plastic strain, $\epsilon_{T-P-MAX}$, always lower or equal to than the maximum strain attainable by the dilatometer. Note that the values of Md^* and Bd^* depend on the value of $\epsilon_{T-P-MAX}$.

4. Results and Discussion

In order to study the thermodynamic conditions under which stress and strain induced transformations occur, the same steel and deformation conditions that were used in Ref. [43] have been replicated in this work. As anticipated, some of the thermodynamic calculations that have been made are specific for the ausforming temperatures $T_{def} = 520\text{ }^{\circ}\text{C}$, $400\text{ }^{\circ}\text{C}$ and $300\text{ }^{\circ}\text{C}$.

4.1. Critical temperatures predictions

4.1.1. Stress-free strain-free transformations

For a stress-free strain free-state, Figure 3 (a) represents both $\Delta G^{\gamma \rightarrow \alpha}$ and $\Delta G^{\gamma \rightarrow \gamma' + \alpha}$ as a function of temperature. Note that the bainitic nucleation is possible all over the temperature range, so the most limiting condition is its growth. The M_s and the B_s temperatures have been calculated as previously explained and they are 279 and $555 \pm 42^{\circ}\text{C}$, respectively, see Table 2. The table also gathers the remaining critical temperatures calculated in the following sections.

Figure 3. Evolution of the driving force for the transformation of austenite to martensite/bainite, showing the calculated critical temperatures. Those

critical temperatures refer to: (a) those describing transformations in which only thermal and compositional changes are considered, i.e. critical temperatures describing stress and deformation free phase transformations; (b) those describing transformation in which stresses below the yield strength are applied, i.e. stress induced transformations; and (c) those describing transformation in which stresses above the yield strength are applied, i.e. strain induced transformations. The shadowed areas show the variability of the curves because of the variability of s , ζ and σ_Y and the variability of ΔG_{SB} .

Table 2. Theoretically calculated critical temperatures for the studied steel. They uncertainty corresponds to the standard deviation of all results obtained by varying the value s , ζ , ΔG_{SB} (just in the case of bainite) and σ_Y (only for $M_s(\sigma)$ and $B_s(\sigma)$) as previously described.

Critical temperature	Calculated value /°C
M_s	279
$M_s(\sigma)$	302±3
M_d^*	349±12
B_s	555±42
$B_s(\sigma)$	584±36
B_d^*	666±53

4.1.2. Stress-induced transformations

Table 3 gathers the predictions of σ_Y by the model of Ref. [76], besides the experimental σ_Y values, estimated by the 0.2 % criterion. It can be found that, although the experimental values are slightly lower than the predicted ones, there are no significant differences when considering the model error, which suggests that no stress- induced transformation has taken place.

Table 3. Comparison of experimentally estimated yield strength at different temperatures with the yield strength values predicted by Ref. [77]

T_{def} /°C	Predicted σ_Y according to Ref.	Experimental
	[77]	σ_Y
	/MPa	/MPa
520	197±17	192±7
400	229±20	213±5
300	257±23	234±6

Figure 3(b) shows the evolution of the total driving force for the transformation as a function of temperature, considering two different mechanical driving forces depending on the phase that is being induced. The total driving forces for the transformation considering the mechanical driving force associated to the martensitic and the bainitic transformations are , $\Delta G^{Y \rightarrow \alpha'}$ and $\Delta G^{Y \rightarrow \alpha_B}$, respectively. By intersecting those two lines with their corresponding threshold values, i.e. $\Delta G_N^{\alpha'}$ and ΔG_{SB} for martensite and bainite, respectively, one can obtain both $M_s(\sigma)$ and $B_s(\sigma)$. Therefore, according to the energy balances, while martensite stress induced transformations can occur up to $302 \pm 3^\circ\text{C}$, in good agreement with the results obtained by comparing the model with the experimental σ_Y as the model started not to fit the experimental curve at the yield point, bainite stress induced transformations can be found up to $584 \pm 36^\circ\text{C}$.

4.1.3. Strain-induced transformations

Firstly, in order to prove that the deformation mechanism is TRIP, the data-driven machine learning technique developed by Chaudhary et al. [92] has been used. Although it does not output a value of Stacking Fault Energy (SFE), it classifies the steel at room temperature into three different categories

correspondent to the mechanical behavior that has been predicted, according to the SFE: TRIP-like behavior ($\text{SFE} < 20 \text{ mJ/m}^2$), Twinning-Induced-Plasticity (TWIP)-like behavior ($\text{SFE} = 20 - 45 \text{ mJ/m}^2$) and dislocation glide ($\text{SFE} > 45 \text{ mJ/m}^2$). According to this technique, the steel subject of study behaves in a TRIP-like way at room temperature so, as SFE increases with temperature in austenite [93], it is possible for TRIP transformations to occur at higher temperatures, although it is also possible that the mechanism changes to TWIP or even dislocation glide, depending on the SFE value.

Secondly, the JC model has been fitted, compared to the experimental curves and used to calculate the corresponding critical temperatures. The temperatures T_r and T_m have been set as 25 and 1497 °C respectively. The experimental values of σ_Y , once it has been confirmed that they haven't been lowered because of stress-induced transformations (see previous subsection), have enabled to obtain the parameters A and m: 282 MPa and 1.03, respectively. The value which m takes, close to the unity, describes the experimentally observed linear dependence of the yield strength with temperature which, although not common, has been previously observed in austenitic steels [26, 94]. To fit B and n, only the curve corresponding to the highest temperature (520°C) up to 25 % has been used. The reason of this decision is that it presents a small deviation for strains higher than 30%, whereas the curves corresponding to the two lowest temperatures (300 and 400°C) have inflection points at rather low true plastic strains, approximately 12 and 0%, respectively, as shown in *Figure 1(b)*. B and n have been fitted to be 3407 MPa and 0.89, respectively. The term which assesses the effect of the strain rate in the model can be ruled out of the expression (and

thus, C does not have to be fitted) if the parameter $\dot{\varepsilon}_{p0}$ is equaled to $\dot{\varepsilon}_p$. In addition, the value $\dot{\varepsilon}_p = \dot{\varepsilon}_{p0} = 0.04 \text{ s}^{-1}$ can be considered a quasi-stated strain rate, which was recommended by Schwer [95] in order to obtain reliable results. All fittings have been done by using the Nelder-Mead method [96] implemented by Matlab® to minimize the error with respect to the engineering experimental curves. Note that these engineering stress-strain curves must be finally converted to true stress-strain for the calculation.

The comparison of the prediction with the experimental values has been made in terms of the true stress (σ_T) - true plastic strain (ε_{T-P}) curves, which can be found in Figure 4.(a), (b) and (c) and in terms of strain hardening rate $d\sigma_T/d\varepsilon_{T-P}$, see Figure 4 (d), (e) and (f). Whereas no perceptible changes can be seen for large strains and $T_{\text{def}}=520 \text{ }^\circ\text{C}$, suggesting that if there has been any phase transformation during that step, its volume fraction must be rather low, deviations are much clearer for the other two temperatures, especially for the lowest one (300°C), for which the prediction does not fit the data at all. While the typical behaviour of the theoretical strain hardening rate is steady decreasing, for the case of $T_{\text{def}} = 400^\circ\text{C}$ $d\sigma_T/d\varepsilon_{T-P}$ it starts to increase from 12% onwards. In the case of $T_{\text{def}} = 300^\circ\text{C}$ this response is even more marked, as the prediction of $d\sigma_T/d\varepsilon_{T-P}$ does not overlay the experimental lines at any point, suggesting that the transformation has started right after the yield point has been achieved. Those deviations have been shown to be related to the formation of α' in other studies (strain induced martensite) [9, 22, 23, 25, 26], although the explanation would be homologous in case strain induced bainitic transformations would have happened.

Figure 4. Comparison of experimental results (lines) vs predictions (dots) obtained by JC model, where σ_T and ϵ_{T-P} stand for the true stress and true plastic strain, respectively, whereas $d\sigma_T/d\epsilon_{T-P}$ is the strain hardening rate.

Once the fitting is finished, $\Delta G^{\gamma \rightarrow \alpha}$ can be calculated as a function of ϵ_p for bainite and martensite, $\Delta G^{\gamma \rightarrow \alpha'}$ and $\Delta G^{\gamma \rightarrow \alpha_B}$, respectively. As already anticipated, due to the dilatometer technical limitations (maximum plastic true strain of 35 %), the JC model can only be verified up to that level. Therefore, energies have only being calculated up to that value, which has also being decided to be $\epsilon_{T-P-MAX}$, see Section 3.3. The obtained results are presented in Figure 5. Note that the $\Delta G^{\gamma \rightarrow \alpha'}$ differs from $\Delta G^{\gamma \rightarrow \alpha_B}$ because the mechanical driving force, ΔG_{mech} (Equation (7)) is different depending on the dilatational and shear components of the shape change, s and ζ , respectively. From the figure, it is clear that only for $T_{def}=300^\circ\text{C}$, strain induced martensitic transformation is possible all over the range of study. The transformation starts right after σ_Y is achieved and it keeps going as long as deformation is applied, in good

agreements with the results obtained by the JC model, see Figure 4(c). However, for higher temperatures (400 and 520°C), $\Delta G^{\gamma \rightarrow \alpha'}$ and $\Delta G^{\gamma \rightarrow \alpha_B}$ are always lying in the range between $\Delta G_N^{\alpha'}$ and ΔG_{SB} , which means that the system energy is never high enough in absolute value for the martensitic transformation to occur, although it is high enough for strain induced bainitic transformations to happen. Bear in mind that, even though the strain induced bainitic transformations are thermodynamically possible at those temperatures for any strain level, the bainitic transformation takes time, so it is possible that it starts after some time, when already higher strain levels have been attained, as suggested by the comparisons of the JC model and the experimental results.

From the calculations at the different T_{def} in Figure 5, the values of $\Delta G^{\gamma \rightarrow \alpha}$ for $\varepsilon_{T-P-MAX}=35\%$ have led to the calculation of the critical temperatures Md^* and Bd^* , see Figure 3(c). Note that the bainite nucleation is possible all over the temperature range for a stress-free strain-free state, as previously mentioned. As plastic deformation promotes the bainitic nucleation, it can be ensured that growth is the most limiting condition for the present steel. After applying the conditions shown in Equations (9) and (11), it can be said that the Md^* and Bd^* temperatures are 349 ± 12 and $666 \pm 53^\circ\text{C}$, respectively.

4.2. Experimental Evidences

4.2.1. Reference treatments

First, to obtain a microstructure composed mainly of martensite and a small amount of austenite, the steel has been austenitized at 990°C for 240 s and then cooled down to room temperature at 15°C/s , see treatment C in Figure 1(a).

Such cooling rate was proved to be high enough so that no other transformations interfere during cooling, as proved by dilatometry. The experimentally estimated Ms temperature, 288 °C is in good agreement with the thermodynamic equations, which predicted that the Ms would be 279 °C.

Figure 5. Calculated driving forces $\Delta G^{\gamma \rightarrow \alpha}$ (Equation (11)) for martensite ($\Delta G^{\gamma \rightarrow \alpha'}$, solid line) and bainite ($\Delta G^{\gamma \rightarrow \alpha_B}$, dashed line) during compression tests as a function of the applied plastic strain and at different T_{def} . The horizontal lines represents the $\Delta G_N^{\alpha'}$ and ΔG_{SB} values from Equations (1) and (4) respectively. The shadowed areas show the variability of the curves because of the variability of s and ζ and the variability of ΔG_{SB} .

Second, to obtain a bainitic microstructure, and following the scheme I in Figure 1(a), the steel was cooled down to different T_{ISO} , i.e. 520, 400, 350, 325 and 300 °C, and held isothermally (no deformation applied) for 1 hour before cooling down to T_{room} . During isothermal holding, Figure 6(a), dilatometry only revealed bainitic transformation at T_{ISO} lower than 400 °C, after which no martensitic transformation was detected on cooling. However, as $T_{\text{ISO}} = 520^\circ\text{C}$ is in the bay between the ferritic/pearlitic region and the bainitic region, no bainitic

transformation was detected (no relative change in length detected during the whole experiment, see Figure 6(a), and a martensitic transformation was detected on cooling, whose experimental M_s is similar to the one obtained for the as-cooled experiment, i.e. 293 °C. The absence of bainite for such temperature is also in good agreement with the thermodynamic calculations, which stated that B_s is 553 °C.

Figure 6. Results on pure isothermal treatments, sketch I in Figure 1(a): (a) Dilatometric signal obtained during the isothermal step; T_{ISO} stands for the temperature at which the isothermal treatment was performed. The shadowed area represents the the maximum time (t_{max}) taken by the deformation step, including both short holding times, encircled region in Figure 1(a), and cooling down to T_{ISO} .; (b) Hardness HV10 and plate-like ferrite volume fractions, V_{α} , obtained after pure isothermal treatments. The HV values are compared to the one obtained by treatment C in Figure 1(a), represented by a dashed line.

Figure 7 gathers the micrographs of the microstructures obtained for the previous experiments, whereas their corresponding hardness values and plate-

like ferrite volume fractions can be found in Figure 6(b). Both, the microstructures of the as-cooled material and the one obtained isothermally treating the steel at $T_{\text{ISO}} = 520^{\circ}\text{C}$, show comparable microstructures with very similar HV values. The bainitic microstructures obtained for $T_{\text{ISO}} = 400^{\circ}\text{C}$ are differentiable from the martensitic microstructures because of their hardness levels, which are about 150 to 300 HV10 lower than the hardness of the as-cooled sample. These HV10 values increase as the isothermal temperature decreases, which is most likely related to the increase in ferrite volume fraction, see Figure 6(b), and to a possible refinement of the microstructure, in accordance with other many studies on this type of microstructures [97-100]. Finally, rather big blocks of retained austenite can be seen for the sample that was isothermally treated at $T_{\text{ISO}} = 400^{\circ}\text{C}$, whereas the size of these blocks decreases for lower T_{ISO} .

Figure 7. Micrographs of the microstructures obtained after the reference heat treatments: (a) Martensitic microstructure obtained by

austenitization and cooling to room temperature, treatment C in Figure 1(a); (b-f) Micrographs showing the microstructures obtained by pure isothermal treatments, I in Figure 1(a), with T_{ISO} equal to 520, 400, 350, 325 and 300 °C, respectively.

4.2.2. Single hit compression tests

In order to test the hypothesis of strain induced transformations during the deformation step, additional treatments were performed, see sketch D+C in Figure 1(a). Samples were austenitized and cooled down to T_{def} , at which single hit compression tests were carried out at two different strains per temperature, using a strain rate equal to 0.04 s^{-1} . A strain of 10 % and a strain which is close to the maximum attainable strain by the equipment at the given temperature ($\epsilon = 30, 20$ and 15% for $T_{\text{def}}=520, 400$ and 300 °C , respectively), were selected to study the limit cases. After deformation, samples were unloaded and temperatures were held for 10 s, see inset in Figure 1(a). This step was followed by a final cooling down to room temperature, where only a martensitic transformation was detected, see Figure 8(a) for estimated M_s temperatures during such coolings. Taking into account the deformation conditions, the holding times before and after the compression step and the cooling conditions to room temperature, the maximum time that can go by since samples reach T_{def} until they are deformed and cooled down to room temperature, t_{max} , is short enough not to exceed the incubation time of the bainitic transformation at any of the tested temperature, see Figure 6(a). Therefore, any distinguishable bainite present in the martensite formed on cooling has been formed during the deformation step. The hardness values of the samples treated this way can be found in Figure 8(b).

Figure 8. (a) M_s temperatures obtained during final cooling to room temperature and (b) hardness values HV_{10} of the final microstructures. All values correspond to the single hit compression tests, D+C in Figure 1, except the dashed line, which corresponds to the reference M_s/HV_{10} , obtained for the as cooled material, treatment C in Figure 1.

Figure 9 shows the microstructures obtained after the mentioned single hit compression tests. For the highest temperature and the lowest strain, i.e. 10% at 520 °C, Figure 9(a), an homogeneous martensitic microstructure, characterized by a similar hardness to the one obtained for the as-cooled material, is observed, see Figure 8(b), confirming that no bainitic transformation took place. Furthermore, the M_s obtained during cooling to room temperature, see Figure 8(a), is the same than the one of the as-cooled material, showing that there were no microstructural changes before the cooling step; note that the applied strain seems not to have significant influence on the bulk M_s in this case. For higher strain levels, even though the true stress-true strain curves did not show any evidence which would have made think that there had been transformation during

the deformation step, see Figure 4(a), a different phase can be detected among the martensite, see Figure 9(b). This disagreement can mean that small fractions of a strain induced phase do not modify the curve significantly. A higher magnification micrograph indicates that this phase is granular bainite, as it is apparently coarser and some blocks of MA constituent can be identified in it. A reduction of almost 80 HV10, in comparison with the as-cooled microstructure and the one corresponding to $\varepsilon = 10\%$, confirms that there is presence of softer phases than martensite in the microstructure. In addition, the M_s temperature estimated during cooling to room temperature is lower than the one obtained for lower plastic strains and for the as-cooled condition, which is most likely due to the combination effect of the mechanical stabilization of austenite and the thermal stabilization of this phase because of the C-enrichment associated to the bainitic transformation.

For both treatments performed at $T_{\text{def}}=400\text{ }^{\circ}\text{C}$, bainite can also be detected among the martensitic microstructure, although in this case the bainitic phases consists of long plates, as opposed to the previous cases, see Figure 9 (c) and (d). The detected reductions in hardness are in good agreement with the microstructural characterization, being the hardness lower for the case in which a higher bainite fraction is detected, i.e. $T_{\text{def}} = 400\text{ }^{\circ}\text{C}$ and $\varepsilon = 20\%$. The M_s temperatures estimated during cooling to room temperature for both treatments are the lowest ones among all estimated M_s for these single hit compression tests, even lower than the ones obtained after deforming at $T_{\text{def}} = 300\text{ }^{\circ}\text{C}$, temperature at which the austenite is prone to be more mechanically stable, suggesting that the formation of bainite has played an important role when it

comes to this parameter. The presence of bainite for such deformation temperature supports the assumption of the accuracy of the calculation of M_d^* by thermodynamic calculations for fully austenitic microstructures.

Finally, no bainite has been detected on the micrographs corresponding to $T_{\text{def}} = 300\text{ }^{\circ}\text{C}$, regardless the applied deformation, see Figure 9 (e) and (f), and the microstructure is a mixture of martensite formed during the deformation step and fresh martensite formed on cooling to room temperature. The estimated M_s temperatures are lower than the ones obtained for the as-cooled material, indicating that a certain degree of austenite stabilization is taking place and more austenite is expected in the microstructures. However, the hardness levels are almost 20 HV10 higher, which suggests that the increase in dislocation density is playing a role. Therefore, it is confirmed that, during the deformation step at the lowest temperature, austenite transforms to martensite rather than to bainite, in good agreement with the performed theoretical calculations.

Figure 9. Micrographs showing the microstructures obtained after performing single hit compression tests and cooling down to room temperature, treatment D+C in Figure 1(a). In the tests, different deformations ϵ have been applied at different temperatures T_{def} . (a) 10 % at 520 °C; (b) 30 % at 520 °C; (c) 10 % at 400 °C; (d) 20 % at 400 °C; (e) 10 % at 300 °C; and (f) 15 % at 300 °C.

4.2.3. Ausforming treatments

The effect of the observed strain induced bainitic transformations on the final microstructures obtained by ausforming treatments, see D+I in Figure 1(a), has been also studied. The ausforming treatments consisted of a full austenitization, followed by a cooling down to T_{def} . These steps were homologous to the ones of the previous studied treatments. The selected T_{def} are 520 and 400 °C, as both lead to strain induced bainite. After that, samples were deformed up to 10 % and a strain close to the maximum attainable strain at the given temperature, i.e. 20 and 30 % for $T_{\text{def}} = 520$ and 400 °C, respectively, all of them studied in the

previous subsection. Subsequently, samples were cooled down to two different T_{ISO} , i.e. 325 and 350 °C, which were held for 1 hour before cooling down to room temperature. During this holding, anisotropic dilatometry signals characteristic of these ausforming treatments were detected [43].

The microstructures obtained by ausforming treatments with $T_{ISO} = 325$ °C along the transverse section can be observed in Figure 10. All microstructures are bainitic, although the microstructure obtained after deforming at 520 °C up to 30 % are coarser, as they are formed by plate-like bainite formed during the isothermal step and by the granular bainite formed during the compression step. Some apparent alignment can be detected for the samples ausformed at 400 °C, in concordance with previous studies [4, 43, 101]. Note that all the discussion has been done for $T_{ISO} = 325$ °C as the morphologies obtained by these treatments and the trends observed for other parameters that are subsequently described are the same than the ones obtained for $T_{ISO} = 350$ °C with the only difference that plates transformed during the isothermal holding are slightly coarser, so the influence of the presence of phases transformed during the compression step is less noticeable.

Figure 10. Micrographs showing the microstructures obtained after treatments D+I in Figure 1(a), where T_{ISO} is in all cases 325 °C, whereas T_{def} and ϵ are, respectively: (a) 520 °C and 10 %; (b) 520 °C and 30 %; (c) 400 °C and 10 %; and (d) 400 °C and 20 %.

With respect to the volume fraction of bainitic ferrite, it can be noted that applying deformation to the austenite mechanically stabilizes it, see Figure 11(a), as reported by many other authors [43, 80, 81, 102-104]. Note that the microstructure is only composed of ferrite and austenite, as confirmed by XRD. However, it can be seen that it is mainly the strain which affects the bainitic ferrite volume percentage, as such values seem to remain unaffected to the deformation temperature.

The bainitic ferrite plates have been measured on both the transverse and the longitudinal sections, see Figure 11(b-c), and there are several issues to mentioned: a) as was pointed out in Ref. [43], when the bainitic transformation is anisotropic, the stereographic correction which is used to obtain the final average

plate thickness value might not be correct, as the applied method and correction and thought for isotropic microstructures. For that reason, important differences arise between the plate thickness values obtained from transverse and from longitudinal sections; b) the differences that are discussed in the next point are usually more pronounced when the isothermal temperature is the lowest one; and c) for $T_{\text{def}}=520^{\circ}\text{C}$, temperature for which almost no strain induced bainite had been detected, the plate thickness values are always lower than the reference one, regardless the deformation level. In addition, it can be seen that, generally, larger deformations promote a more effective refinement, even though granular bainite is present. For $T_{\text{def}}=400^{\circ}\text{C}$, however, the obtained microstructures are generally not refined with respect to the reference plate thickness and even in some cases, the average plate thickness is coarser than the one measured for the isothermal treatment, most likely because of the strain induced plate like bainite formed at higher temperatures.

Finally, concerning the samples Vickers hardness, it can be seen that applying low levels of deformation at $T_{\text{def}}=520^{\circ}\text{C}$ enables to maintain the same hardness level with respect to the reference state, see Figure 11(d), because of the microstructure refinement, even though the amount of bainitic ferrite which is present in the microstructure is lower, see Figure 11 (a). Note that the same effect was observed for the single compression tests performed at 520°C up to 10%. However, the austenite mechanical stabilization, together with the presence of granular bainite, provoke that the sample deformed at $T_{\text{def}}=520^{\circ}\text{C}$ up to the highest deformation level is softer than the reference one, even though a refinement has been generally observed. Regarding the samples deformed at

400 °C, both present lower hardness values than the reference state, because of the reduction in bainitic ferrite volume percentage and in some cases the increase of plate thickness, therefore evincing the fact that the presence of phases formed during the compression step can impair the final mechanical properties.

Figure 11. Comparison of the ausformed samples with the pure isothermal microstructures in terms of (a) ferrite volume percentage, V_{α} ; (b) plate thickness average measured throughout the transverse section; (c) plate thickness average measured throughout the longitudinal section; and (d) Vickers Hardness (10 kg), HV10.

5. Conclusions

All critical temperatures describing the temperature ranges in which stress and strain-free, stress-induced and strain bainitic or martensitic induced phase transformation can occur have been calculated by applying a new theoretical approach based on thermodynamic calculations.

Evidences of martensitic and bainitic strain induced transformations have been experimentally revealed at temperatures at which they were predicted, showing the validity of the proposed theoretical approach for the determination of those critical temperatures.

The application of deformation during ausforming treatments has shown to decrease the amount of bainitic ferrite due to the austenite mechanical stabilization phenomenon. Even though, in the cases in which no strain induced phases were detected, a refinement was observed, enabling to keep the same hardness levels than for isothermal treatments. However, the presence of phases formed during the compression step, i.e. granular bainite or coarse plate-like bainite, during such treatments, has been shown to be detrimental, increasing the average plate thickness and reducing the hardness values in comparison with the reference microstructures.

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