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Evolution of austenite static recrystallization and grain size during hot rolling of a V-microalloyed steel

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ABSTRACT

Laboratory double-deformation isothermal tests and multipass continuous cooling hot torsion tests were used to study the static recrystallization of austenite under isothermal and anisothermal conditions as well as to simulate the hot rolling of a 0.13% V-microalloyed steel. Characterization of the evolution of austenite microstructure was carried out. It has been verified that no-recrystallization temperature (T_{nr}) approximately corresponds to the temperature where recrystallization starts to be incomplete during rolling. However, incomplete recrystallization is visually evident at temperatures 25–50 °C below T_{nr}, where grain elongation and increase in aspect ratio with temperature drop start to be significant. An accurate method to estimate the recrystallized fraction during hot rolling from stress–strain data and with no need of metallographic studies has been designed. The results of this method have been compared to metallographic measurements, the values of anisothermal fractional softening and the accumulated stress measured in the MFS plots at $T < T_{nr}$. A pronounced austenite grain refinement has been detected in the first hot rolling passes after reheating, as grain size decreases from 155 μm to 27 μm in six passes. If the effect of grain size on recrystallization and precipitation is taken into account, the correlation of isothermal and continuous cooling tests as well as the relationship between SRCT and T_{nr} or RLT temperatures can be better understood.

1. Introduction

The static recrystallization of microalloyed steels after hot deformation can be obstructed by the pinning effect exerted by strain-induced precipitates. In the curves that represent the recrystallized fraction against post-deformation isothermal holding time ($X_a(t)$), the inhibition of recrystallization by precipitates is manifested by the formation of plateaus that temporarily interrupt the typical sigmoidal shape of an Avrami's law [1–3]:

$$X_a = 1 - \exp\left(-\ln\ 2\left(\frac{t}{t_{0.5}}\right)^n\right) \quad (1)$$

The exponent n usually takes values between 1 and 2 [1,4,5] and according to some authors it depends on temperature [6–8]. $t_{0.5}$ is the time corresponding to a recrystallized volume fraction of 50%, which depends on temperature (T), equivalent pass strain (ε), strain rate ($\dot{\varepsilon}$), austenite grain size (D) and chemical composition of steel according to [9]:

$$t_{0.5} = A\varepsilon^p \dot{\varepsilon}^q D^s \exp\left(\frac{Q}{RT}\right) \quad (2)$$

where Q is the apparent activation energy, R is the universal gas constant (8.3145 J mol^{-1} K^{-1}) and p, q, s and A are constants.

The temperature limit between the stages of full recrystallization and beginning of recrystallization inhibition as a consequence of precipitation comes at a point known as Static Recrystallization Critical Temperature (SRCT) [9,10]. On the other hand, in a continuous cooling multipass thermomechanical test, the temperature below which the recrystallization of austenite during the interpass time between successive passes starts to be incomplete is known as temperature of no-recrystallization (T_{nr}) [2].

The microstructure of austenite just before cooling to room temperature has a major influence on final ferrite microstructure [11,12]. Therefore, one of the most important aspects to be studied is the accurate assessment of the strengthening state of austenite and the quantification of the volume fraction of recrystallization during rolling at temperatures below T_{nr} and especially at the end of rolling, near A_{r3}. To carry out this characterization, metallographic studies can be used [13,14], but metallography is a time-consuming and not always successful technique. Several mathematical models have been developed for the study of recrystallization or soften-

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ing kinetics during hot rolling of steels, i.e. under non-isothermal conditions [15,16]. The "anisothermal fractional softening" method [17,18] gives an approximation of the recrystallized fraction, but this technique includes the contribution of recovery to softening, which can be very high under certain deformation conditions and/or material characteristics [19]. Besides, it is sometimes necessary to know other empirical constitutive parameters or to make preliminary tests to approximate the yield stress of a fully recrystallized material [18,20].

Niobium is known to be the microalloying element that most delays static recrystallization kinetics, even when it is in solution in the austenite [1,21]. The influence of vanadium (dissolved or precipitated) on static recrystallization is weaker but still significant, as can be seen in the increase in the values of $t_{0.5}$, activation energy and SRCT for higher V additions [7,22–24]. A strong solute drag effect of V on the kinetics of metadynamic recrystallization has also been described [24]. In this work, the evolution of static recrystallization of austenite is characterized in a vanadium microalloyed steel. Thermomechanical tests are carried out under isothermal and continuous cooling conditions and conclusions about the relationship between the results coming from both thermal paths are extracted. An empirical method to estimate the recrystallized fraction during rolling from the stress–strain data obtained in the thermomechanical tests is presented and comparison with the results of metallography and the technique of anisothermal fractional softening (FS) is done.

2. Experimental procedure

The steel studied was manufactured by Electroslag Remelting (ESR) in a laboratory unit capable of producing 30 kg ingots. This technique avoids macrosegregation, both in alloying elements and impurities, and there is considerably less microsegregation. These effects are often present in conventional ingots and continuous casting billets. As Table 1 shows, the steel has 0.48% carbon content and 0.13% vanadium and contains a residual Ti content.

Double-deformation isothermal tests as well as multipass hot rolling simulation tests were carried out in a computer-controlled hot torsion machine, on specimens with a gauge length of 50 mm and a diameter of 6 mm. Prior to the torsion tests the specimens were austenitized during 10 min at 1200 °C, a temperature high enough to completely dissolve the V precipitates [25]. However, it should be taken into account that, despite the very low Ti content, a certain amount of undissolved TiN can remain after reheating, as this type of precipitate is very stable [26]. The calculated temperature for its complete solution in austenite is 1368 °C. The calculated solubility temperatures and the reheating conditions are shown in Table 2.

After reheating, the temperature was rapidly lowered to that corresponding to the first deformation pass in each test. In the isothermal double-deformation experiments, this temperature was maintained after deformation during a certain holding time, after which a longer second deformation was applied in order to calculate recrystallized fraction (X_a). The value of X_a was measured using the "back extrapolation" technique [27]. This method presents the advantage of determining the recrystallized fraction instead of the softened fraction, i.e. the effect of recovery is excluded from the double-deformation data [13], so more accurate comparison with observed microstructures can be carried out.

Temperatures of 950 °C, 1000 °C, 1050 °C and 1100 °C and several holding times between 1 s and 500 s were used in order to build the curves of recrystallized volume fraction against time ($X_a(t)$) as well as the Recrystallization–Precipitation–Time–Temperature (RPTT) diagram [7,28–30], where the interaction between these two processes is clearly observed. The equivalent strain (ε) applied in the first pass was 0.20, lower than the critical strain which leads to the start of dynamic recrystallization. The strain rate ($\dot{\varepsilon}$) was kept at 3.63 s^{-1}.

In some samples the second deformation after holding time was replaced by a water-quench and subsequent metallographic preparation. Microstructure was observed in optical microscope to verify the accuracy of back extrapolation method in determining the recrystallized fraction and to compare with the results of multipass simulations. All the microstructural studies carried out in this study were done observing more than 20 fields on a longitudinal surface of the specimens at 2.65 mm from the axis. To reveal the prior austenite grain boundaries, the samples were etched with an aqueous solution of saturated picric acid mixed with a wetting agent. Some droplets of hydrochloric acid were added just before etching to activate the solution.

In the hot rolling simulation tests, the temperature of the first pass was 1150 °C. The simulations consisted of 20 passes made under continuous cooling conditions, with a temperature step of 25 °C between passes, the last pass being carried out at 675 °C. The time between successive deformations, known as "interpass time" (Δt) was equal to 20 s. The strain and strain rate applied in each pass were the same as those used in isothermal tests ($\varepsilon = 0.20$, $\dot{\varepsilon} = 3.63\ s^{-1}$). After determining the critical hot rolling temperatures (T_{nr} and A_{r3}), supplementary quench-interrupted tests were carried out in order to evaluate the evolution of microstructure (grain size and recrystallization) during rolling. In these tests, samples were deformed following the same schedule until they were water-quenched from certain temperatures along rolling schedule. In most cases the sample suffered a last deformation step and then the temperature was lowered 25 °C during the corresponding Δt to reach the quenching temperature, so microstructure just before the application of the rolling pass was assessed. In some case, the sample was quenched immediately after deformation to observe the microstructure prior to a hypothetical cooling from a certain last hot rolling-pass temperature. The austenite grain size (D_γ) and aspect ratio were determined by means of the linear intersection technique. Recrystallized fraction was measured using standard point-counting metallographic technique. Distinction of recrystallized and deformed grains was based on the shape and size of the grains as well as the appearance of grain boundaries [19,31].

Table 1
Chemical composition of the steel studied (wt.%).

C	0.48
Si	0.28
Mn	1.45
P	0.024
S	0.018
Al	0.009
Cr	0.22
V	0.13
Ti	0.003
N	0.0200
O	0.0044

Table 2
Calculated solubility temperatures of the steel studied [25]. Reheating temperature (reheating time = 10 min) and measured austenite grain size (D_γ) at reheating temperature.

Solubility temperatures (°C)				Reheating temperature (°C)	D_γ (μm)
VN	$VC_{0.75}$	TiN	TiC		
1141	904	1368	979	1200	155