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Materials Research and Technology www.jmrt.com.br

Recrystallization of niobium stabilized ferritic stainless steel during hot rolling simulation by torsion tests

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ARTICLE INFO

Article history: Received 10 July 2015 Accepted 16 July 2015 Available online 25 August 2015

Keywords: Rolling simulation Ferritic stainless steel Recrystallization Niobium Ridging

a b s t r a c t

The aim of this study was to investigate the effect of finishing hot rolling temperature in promoting interpass recrystallization on a Nb-stabilized AISI 430 ferritic stainless steel. Torsion tests were performed in order to simulate the Steckel mill rolling process by varying the temperature ranges of the finishing passes. Interrupted torsion test were also performed and interpass recrystallization was evaluated via optical microscopy and electron backscatter diffraction (EBSD). As a result of this work, it has been established, within the restrictions of a Steckel mill rolling schedule, which thermomechanical conditions mostly favor SRX.

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[http://dx.doi.org/10.1016/j.jmrt.2015.07.003](dx.doi.org/10.1016/j.jmrt.2015.07.003)

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1. Introduction

Ferritic stainless steels are an interesting alternative to austenitic stainless steels, owing to their excellent properties even with only small contents, or absence, of nickel. As such, there is increasing interest in developing and improving ferritic stainless steels by industry. These types of steels, like the AISI 430, are widely used in applications that require deep drawing, such as kitchen tools, sinks, and appliances; therefore, surface finishing is very important [\[1\].](#page-7-0) However, these steels are known to present scratches like defects at the surface, during drawing. This phenomenon, referred to as ridging or roping is deleterious to surface quality and must be decreased if not eliminated. The presence of this defect at surface is related to the crystallographic texture of the steel [\[1,2\].](#page-7-0)

After solidification, this type of steels present mostly {0 0 1}<uv0> oriented columnar grains, which is generally considered harmful $\left[3\right]$. It is well known that cold rolled ferritic stainless steels inherit its texture from hot rolling [\[4\],](#page-7-0) which in turn inherit its texture from solidification. In order to decrease ridging this texture must be somehow changed. This can be achieved via either phase transformation or mechanical straining and recrystallization. However, Nb-stabilized AISI 430 ferritic stainless steel, the object of the present study, does not transform on cooling. Therefore, the only possibility to change solidification texture after thermomechanical processing is by recrystallization $[5,6]$. In the case of ferritic stainless steels, it is well known that dynamic recovery (DRV) is the dominating softening mechanism $[7-9]$. This mechanism reduces the stored energy from deformation, hence decreasing the driving force for static recrystallization (SRX). In order to promote SRX, it is then necessary to deform at temperatures which do not favor DRV [\[5,8\].](#page-7-0)

Industry hot rolling of ferritic stainless steels is performed in two stages: roughing and finishing. During roughing, a slab is reduced in 7 passes from 200mm to 28mm with an exit temperature between 1000 and 1050 $°C$. Finishing can be performed either in a hot strip mill or in a Steckel type mill. In the case of the Steckel mill, the transfer bar is reduced in 1–7 passes, from about 28 to a final thickness in between 2 and 12.7 mm at a temperature range of 950–900 $°C$ [\[5\].](#page-7-0) Industrial hot rolling is a complex process. There is, of course, the possibility of performing industrial scale trials to optimize temperature ranges, which would be more favorable to SRX. However, these trials are expensive, difficult to conduct and usually samples cannot be taken from intermediate passes, thus making it impossible to have details of microstructure evolution during processing. The alternative is to use mechanical testing such as hot compression or hot torsion.

Hinton and Beynon [\[7\],](#page-7-0) for instance, simulated Steckel mill rolling conditions via plane strain compression. They used an AISI430 ferritic stainless steel as experimental material in order to study the restoration mechanisms in their simulation. This steel, however, undergoes phase transformation on cooling which, as just mentioned, favors recrystallization and improves texture regarding ridging. Mehtonen et al. [\[6\]](#page-7-0) also studied softening mechanisms, texture changes, dislocation densities and general microstructure evolution during

deformation of dual stabilized Ti–Nb high Cr ferritic stainless steel. In their case, Steckel mill rolling conditions were simulated using plane strain compression. They applied two passes at relatively high temperatures then cooled the sample down to 800 and 650 ℃ and applied a third deformation. They concluded that full recrystallization occurred between passes at high temperatures and that, as the temperature decreased in the third pass, work hardened was more and more preserved, thus having the potential to improve recrystallization further down in the process, during annealing after hot rolling. In their case, the steel used did not transform on cooling. However, the number of passes given was very limited (just three) and the temperature rather low, compared to the usual temperatures employed in an industry process.

In the current work, hot finishing rolling on a Steckel mill of Nb-stabilized AISI 430 ferritic steel was simulated via hot torsion test. This steel does not also transform on cooling, hence changes in texture can only be promoted from recrystallization. In this respect, it is expected that the experimental steel will behave much like the reported for Ti–Nb steel. However, by using hot torsion, it was possible to simulate conditions much more similar to those found in industry, namely, by applying a larger number of passes than that used by the authors just mentioned. The temperatures used in the experiments were also very close to those found in actual Steckel rolling practices. The aim of the present work is then:

(a) to simulate this process via hot torsion testing and

(b) investigate, within the restrictions of a Steckel hot rolling schedule, which thermomechanical conditions, largely related to pass temperature, would mostly favor SRX.

2. Experimental procedure

2.1. Chemical composition

The steel used in the present work was a Nb-stabilized AISI 430 ferritic stainless steel, hereafter denominated 430Nb steel, with chemical composition as shown in Table 1.

2.2. Thermomechanical parameters for hot rolling

Rolling at a Steckel type mill at Aperam South America steelworks was taken as reference for the hot torsion simulations. Detailed description of the mill is given elsewhere [\[10\].](#page-7-0) Table 2 below gives the industrial data used as base for the simulations.

2.3. Hot torsion simulations

For rolling simulations, 15 mm (length) \times 7 mm (diameter) torsion samples were machined from a 28mm thickness samples taken after roughing rolling. Hot torsion tests were performed in an INSTRON 1125 testing machine equipped with a torque cell 200 Nm maximum capacity. Heating of samples was carried out in induction furnace. Tests were performed in argon atmosphere in order to prevent excessive oxidation of the specimens.

During the tests, the specimens were heated at 2° Cs⁻¹ to 1050 °C, held for 120 s, and then cooled at 1° Cs⁻¹ to the temperature of the first pass. A total of five passes were applied with a constant strain rate of $0.3 s^{-1}$. The values of true strain (ε*eq*) applied to the sample are equivalent to those obtained from industry rolling data and calculated as shown below.

$$
\varepsilon_{eq} = \frac{[2\ln(h_1/h_2)]}{\sqrt{3}}\tag{1}
$$

where h_1 and h_2 are the initial and final thickness of the plate, respectively.

The true strain is converted to the torsion angle by

$$
\theta = \frac{\sqrt{3}L\varepsilon_{eq}}{r} \tag{2}
$$

where θ is the angular displacement, *L* is the length and *r* is the radius of the specimen.

Measured values at torque were converted to true stress using

$$
\sigma_{eq} = \frac{\sqrt{3}\tau(3+m+n)}{2\pi r^3} \tag{3}
$$

where σ_{eq} is the equivalent stress, τ is the measured torque, m is the sensitivity coefficient to angular strain rate and *n* is the sensitivity coefficient to angular strain changes. In the present work, the values used were 0.3. [Table](#page-3-0) 3 shows details of the deformation schedules used in the torsion experiments.

2.4. Optical microscopy

Metallographic analyses were performed on the longitudinal sections of the samples in a plane located 0.1mm below the surface in order to obtain a ∼2mm wide section. The samples were prepared by grinding in the usual way, polishing with $1\,\upmu\mathrm{m}$ diamond, and subsequently, polishing for 1 min with colloidal silica composed of 40nm granules. The sample surface was then immediately depassivated by immersing the sample for 60 s in a solution of 100mL, 2mL, and two drops of distilled water, acetic acid, and colloidal alumina, respectively. Samples were also color-etched at 75 ◦C for 105 s using a solution of 110mL of distilled water, 12mL of H2SO4, 4mL of HF and five drops of HNO₃. An optical microscope with polarized light was used to obtain colored images.

[Fig.](#page-4-0) 1(a) shows schematically the location of the surface where metallography was carried out. The surface corresponds to the θZ plane, where θ is the shear direction and *Z* is the axial direction. [Fig.](#page-4-0) 1(b) shows the appearance of an actual sample after being tested.

2.5. Electron backscatter diffraction – EBSD

For EBSD measurements, the sample was prepared in the same manner as that used for optical microscopy, including polishing with diamond paste. Afterwards, the samples were polished with colloidal silica for 40min. EBSD measurements were performed using a Fei Quanta 650 FEG microscope equipped with an Oxford High Speed Nordlys Max 2 detector. The data were collected using an accelerating voltage of 20 kV, a working distance of 18 mm and step size of 3 μ m. Data were analyzed by the OIMTM software. The mean grain size was measured in order to analyze the refinement of the microstructure. Measurements were obtained from EBSD data considering a misorientation angle ≥ 15◦.

3. Results

3.1. Flow curves

[Fig.](#page-4-0) 2 shows flow curves obtained from simulations performed in hot torsion testing. The simulations were intended to model passes given in the Steckel mill, as pointed out earlier. They can be distinguished from one another by start temperature at first pass and temperature at last pass, as displayed in [Fig.](#page-4-0) 2. Hence 1050–990 ◦C means simulation 1 and 930–870 ◦C, simulation 5.

The level of the stress–strain curves increases as temperature decreases, as expected. The shape of the curves are however very similar in all experiments, that is, all curves indicate the occurrence of recovery as the sole dynamic softening mechanism. When passes were given at higher strains, case of passes 1–3 in which strains were respectively 0.43, 0.62 and 0.56, even then, no dynamic recrystallization happened. In these passes the flow curves reach a plateau, in special in the case of curves seen at higher temperatures (the simulation run at 1050–990 $^{\circ}$ C). This is typical behavior for hot resistance of 430Nb steel. So it can be concluded that, under the conditions tested, the steel was always dynamically softened by dynamic recovery only.

Other important information that can be obtained from the stress–strain curves shown in [Fig.](#page-4-0) 2 is the fractional softening between deformations. The fractional softening can be calculated by

$$
\%S = \frac{\sigma_3 - \sigma_2}{\sigma_3 - \sigma_1}
$$

all symbols shown in [Fig.](#page-4-0) 2(f). This parameter is very often used to calculate fractional softening in the case of hot deformation of austenite [\[11\].](#page-7-0) However, in the case of ferritic steels, it seems, the use of a parameter like this may be not very effective or conclusive, as for the case of austenite. However, it seems, it may be used to indicate trends.

For instance, softening between passes 1 and 2 seems to be high, close to 100% in all simulations, since the shape of the curves are very much alike. Then the level of the stress does not change in the second pass, that is, it stays almost constant suggesting dynamic recovery being very effective in counter balancing strain hardening.

Parameter *S* may not be very effective in pointing out explicitly small differences from pass to pass as in the case of passes 1–3 in all simulations. It seems, however, more effective in pointing out trends, though. In this sense, fractional softening computed between passes 4 and 5 is clearly different from those calculated between passes 1 and 3. In the case of passes 4 and 5 the fractional softening seems smaller than 100%, that is, around 50% or less. Also, dynamic recovery is not effective enough in passes 4 and 5 when compared to previous passes and the shapes of the curves in pass 4 and 5 display some work hardening. Then, it could be concluded here that, although fractional softening may not be a very effective parameter to identify the exact quantities of softening occurring between adjacent passes, it may be used, in the case of 430Nb steel, to identify trends. In this way, the results seem to indicate almost full softening in the group of passes 1 to 3 and some work hardening retained in the group 4-5.

Fig. 1 - (a) Scheme of the surface used for OM and EBSD analysis and (b) sample after torsion.

3.2. Microstructure

[Fig.](#page-5-0) 3 shows the microstructures of samples in the initial, as received condition and those obtained after each torsion simulation. The overall impression is that the initial microstructure is clearly fully recrystallized. The mean grain size is

 $\mathop{\rm approxim}$ ately 100 μ m. After simulation, micrographs indicate almost full work hardened grains. This is consistent with comments just made on the shape of the stress–strain curves and fractional softening calculated from the flow curves. However, optical microscopy does not reveal clearly fine details. For instance, in some areas of micrographs (c) to (f) there

Fig. 2 - Flow stress curves of samples deformed in hot torsion simulating Steckel rolling conditions. Start and finishing temperature were varied 30 °C form one simulation to another: (a) 1050-990 °C, (b) 1020-960 °C, (c) 990-930 °C, (d) 960-900 °C **and (e) 930–870 ◦C. (f) Schematics used in the calculation of fractional softening between passes.**

Fig. 3 - Microstructures of samples (a) prior to torsion simulation and (b) to (f) after last pass of deformation.

might be some small grains among the most elongated ones. This could possibly be due to some incipient recrystallization. As the shape of the stress–strain curves did not indicate any possibility of occurrence of dynamic recrystallization, the alternative is that these small grains might have appeared during cooling after the test. This is supported by similar finding reported in $[8]$. To have a better and finer view as well as to elucidate some doubts on microstructure, EBDS maps were obtained.

[Fig.](#page-6-0) 4 shows EBSD maps revealing more detailed microstructure features. As optical micrographs showed a general trend in the direction of having full work hardened grains at lower temperatures, EBDS maps were performed for samples with torsion simulations finishing at lower temperatures, namely, simulation at temperature ranges of 960–900 ℃ and 930–870 ℃.

As mentioned earlier, torsion simulation allows interruption of test at any stage of the process giving more flexibility to investigate microstructure evolution than industry hot rolling trials. On the other hand, it is impossible for any mechanical test, in particular hot torsion, to replicate an industry process fully. So torsion results must be seen as an approximation to reality and, in this way, the results will probably indicate trends. With this in mind, it is interesting to note that microstructure before pass 3 seems more recrystallized than after pass 5. Recrystallization is essential to change texture in this steel, as already mentioned.

These observations also seem to agree with previous analysis of the shape of flow curves and estimation of fractional softening. That is, fractional softening is larger than 50% for passes 1, 2 and 3 and less 50% for passes 4 and 5. Also the shape of flow curves for passes 4 and 5 were distinctively different from those of passes 1, 2 and 3 indicating some work hardening, remaining after deformation.

As for grain sizes, after pass 5 in the simulation 960–900 ◦C grain size was 85 \upmu m with an aspect ratio of approximately 2:1. After pass 5 in the simulation $930-870$ °C, grain size was smaller as an average, 65 μ m, however the aspect ratio was about the same, 2:1, although there is clear presence of small equiaxed grains, as already mentioned.

Fig. 4 - EBSD maps from torsion samples fast cooled to room temperature prior to pass 3; (a) and (b), pass 4; (c) and (d), pass 5; (e) and (f) and after pass 5; (g) and (h). Samples (a), (c), (e) and (g) were tested in the 960–900°C simulaiton and samples (b), **(d), (f) and (h) in the 930–870 ◦C.**

4. Discussion

As evidenced by the shape of the flow curves [\(Fig.](#page-4-0) 2) and microstructures (Fig. 4), recovery is the main dynamic softening mechanism during the first three passes. The material also softened by static recrystallization between passes, since the calculated softening fractions were approximately 100%. During the last two passes, it was verified that dynamic recovery was not so effective in eliminating fully work hardening since stresses did not achieved a plateau as in the case of the curves for high temperature deformation.

Similar results were observed by Mehtonen et al. [\[6\].](#page-7-0) The authors investigated the flow stress evolution, microstructure, texture, dislocations and precipitation during multi-pass deformation of a dual-stabilized 21%Cr ferritic stainless steel. They observed, under the tested conditions, that the steel softened by dynamic recovery during the first two passes applied at 950 ◦C and recrystallized statically during the interpass time of 20 s. Even though static recrystallization occurred, a small fraction of recovered grains remained in the microstructure, indicating that the static recrystallization was not complete. In their work, only the last pass was applied in a different temperature than the two previous passes. They found that lowering the deformation temperature resulted in a higher amount of work hardening, which may accelerate the SRX kinetics during subsequent hot band annealing stage [\[8\].](#page-7-0)

Results of the present work indicate that full recrystallization occurred at deformations given in passes 1–3 as seen in [Fig.](#page-5-0) 3. The results also indicate partial recrystallization and full work hardening (elongated grains) in the last two passes as also seen in [Fig.](#page-5-0) 3. This is in agreement with results reported in $[6]$, that is, full recrystallization between deformations given at 950 ◦C and work hardening at 800 and 650 ◦C. The latter two temperatures reported in $[6]$, however, are either in the very low end for a Steckel mill processing or altogether unrealistic. In the present work, as torsion was used to simulate the industry process, a more realistic condition could be used hence the results here obtained may approach better those found in the industry.

With the results obtained from this work, it is possible to speculate on which temperature range would be more suitable in order to favor recrystallization and latter work hardening. So it seems that finishing rolling in Steckel mill should be carried out at high temperatures for first passes, allowing the occurrence of recrystallization between passes. Then, final passes should be given at lower temperatures allowing for some work hardening to occur during the last passes.

5. Conclusions

The main results can be summarized as follows:

- (a) Flow curves obtained via torsion test simulation of the finishing hot rolling on a Steckel mill confirmed that interpass recrystallization occurred in the Nb-stabilized AISI 430 ferritic stainless steel at high temperatures deformation. When the simulation temperatures decrease, results indicated partial recrystallization and full work hardening in the last two passes.
- (b) The flow curves and microstructures analysis showed that recovery is the main dynamic softening mechanism during the first three passes for all simulations, while elongated grains showed that work hardening took place in the last two passes performed in the temperature ranges of 960–900 ℃ and 930–870 °C.
- (c) Results reported here may suggest that finishing rolling 430Nb stabilized steels in a Steckel should be carried out in such a way that first passes were given at higher temperature so as to allow static recrystallization to occur between passes. Also, final passes should be applied at lower temperatures so that no static recrystallization would occur between passes and work hardening would occur during deformation so that softening by dynamic recovery would not be so effective as to decrease work hardening significantly.

Conflicts of interest

The authors declare no conflicts of interest.

Acknowledgments

The authors would like to thank CBMM for the financial and technical support, Aperam South America for the technical support and for providing the material for this study, Dr. Marco Antônio da Cunha for the technical discussions, and LNNanno at CNPEM, where the EBSD analyses were performed. FVB and MSA would also like to thank the CNPq (311538/2013-4) and CAPES institutions for academic scholarships and providing financial support during the course of this study.

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