Recrystallization kinetics of austenite in Nb microalloyed steel

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Abstract
The knowledge of the relationship between the thermomechanical conditions and the final microstructure and properties of a steel product, often requires a strong interaction between numerical models and experimental data. In this paper, hot strength and recrystallization kinetics of austenite in a Nb microalloyed steel were experimentally investigated by single and double hot compression tests, varying the strain rates and the deformation temperatures. For comparison a plain carbon steel was studied by the same procedures. The effects of deformation temperature, strain rate and addition of Nb on peak stress and softening/recrystallization fractions of the materials were assessed. The data obtained from the mechanical tests were compared with the results obtained from the metallographic analysis revealing the prior austenitic boundaries and measuring the grain size distribution. Finally, the comparison between the mechanical and metallographic method allowed a better interpretation of the results obtained from the classical double hot compression tests.

Keywords: Recrystallization; double hot compression tests; metallography; hot deformation.

1. Introduction
The modeling and numerical simulation of complex thermo-mechanical industrial processes aiming to the optimization of the steel properties requires a wider knowledge of the evolution of the microstructure at the specific deformation temperature [1, 2]. The coupled thermo-mechanical-microstructural models shall be able to predict the mechanisms restoring the austenite microstructure, i.e. dynamic recrystallization during the deformation at high temperature and static recrystallization in the inter-pass time. The reliability of the final data obtained from the numerical simulation strongly depends on the experimental data on which the models are based [3]. In this paper hot mechanical behaviour and recrystallization kinetics of Nb-microalloyed and Nb-free steels were investigated by single and double hot compression tests, by varying the strain rates and the deformation temperatures. For selected test conditions, the recrystallized grains were observed by metallographic techniques and the results were compared with the ones obtained from the mechanical tests.

2. Materials
Two steels were investigated, P265GH (steel A) and 20MnNb6 (steel B), produced by continuous casting. Chemical compositions are reported in Table 1.

<table>
<thead>
<tr>
<th>code</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P max</th>
<th>S max</th>
<th>Cr</th>
<th>Mo</th>
<th>Ni</th>
<th>Al tot</th>
<th>Cu</th>
<th>Nb</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>&lt;0.20</td>
<td>&lt;0.40</td>
<td>&lt;1.40</td>
<td>0.025</td>
<td>0.020</td>
<td>&lt;0.30</td>
<td>&lt;0.08</td>
<td>&lt;0.30</td>
<td>2 0.020</td>
<td>&lt;0.30</td>
<td>&lt;0.01</td>
</tr>
<tr>
<td>B</td>
<td>&lt;0.22</td>
<td>0.15 to 0.35</td>
<td>1.50</td>
<td>0.025</td>
<td>0.020</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>≤ 0.060</td>
<td>≤ 0.30</td>
<td>≤ 0.015</td>
</tr>
</tbody>
</table>

Table 1. Steels chemical compositions (%wt)

Cylindrical samples were machined from the columnar region of a round billet. Their dimensions were 12.6 mm diameter and 38.1 mm height, according to ASTM E 209-00 Standard [4].

3. Methods
The hot mechanical properties were investigated by single compression tests, whereas the recrystallization kinetics was studied both by double compression tests, varying the inter-pass time between the first and the second compression, and by metallographic techniques. The mechanical tests were performed on a universal electro-mechanical tension-compression Instron testing machine, equipped with a high temperature furnace. In Figure 1 the thermo-mechanical cycle for single compression tests is reported.

Figure 1. Thermo-mechanical cycle of hot single compression tests.
The selected test temperatures and strain rates were respectively 1050°C and 1150°C and 0.01 s⁻¹ and 0.04 s⁻¹. In Figure 2 the double compression test procedure is represented.

**Figure 2.** Thermo-mechanical cycle of hot double compression tests.

The investigated test temperatures and strain rates were the previous ones. The first compression was stopped at the peak stress ($\sigma_2$) previously determined by single compression tests for the same material and the same temperature and strain rates conditions. Then the specimen was unloaded and held at the test temperature for times between 0.5 to 200 s and then reloaded. The softening fraction and the recrystallization kinetics can be evaluated according to equations (1) and (2), respectively. The double deformation method is based on the principle that the yield stress at high temperature can be considered a sensitive measure of the structural changes [3].

$$F_s = \frac{\sigma_p - \sigma_2}{\sigma_p - \sigma_1}$$  \hspace{1cm} (1)

where:
- $\sigma_p$ = peak stress in the first deformation;
- $\sigma_1$ = 0.2% offset stress at first deformation;
- $\sigma_2$ = 0.2% offset stress of second deformation.

$$X(t_i) = \frac{F_s(t_i) - F_{s,\text{recovery}}}{1 - F_{s,\text{recovery}}}$$  \hspace{1cm} (2)

where:
- $X(t_i) =$ recrystallized fraction at inter-pass time $t_i$;
- $F_s(t_i) =$ softening fraction at inter-pass time equal $t_i$;
- $F_{s,\text{recovery}} =$ softening fraction due to recovery assumed equal to 0.2 [3].

Finally, for selected test parameters, the recrystallization kinetics was studied also by metallographic techniques. For this analysis, the austenitic grain size is required and different procedures for revealing and measuring the prior austenitic grain are described in [5, 6]. Béchet-Beaujard etching is the most common method to reveal austenite boundaries in low-alloy steels, but it was demonstrated not to be so effective on the investigated steels. Hence, the pro-eutectoid ferrite nucleation method was considered. Some samples were heated according to the thermal cycle described in Figure 1: after the soaking at the compression temperature, the samples were properly cooled to room temperature in order to nucleate ferrite at austenite grain boundaries. After a Nital 2% etching the LOM (light optical microscope) analysis was carried out by an image analysis software, in order to evaluate the grain size distribution before straining. The same method was used in the case of deformed specimens. Fine and equiaxied grains were identified as recrystallized austenite.

4. Results

**Single compression tests**

Figure 3 shows an example of hot compression stress-strain curves.

**Figure 3.** Typical single compression curves-$T=1150°C$, strain rate 0.04 s⁻¹.

The average peak strains and the average peak stress were evaluated and reported in Table 2.

<table>
<thead>
<tr>
<th>Steel A</th>
<th>1050°C</th>
<th>1150°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.01 s⁻¹</td>
<td>0.24</td>
<td>0.19</td>
</tr>
<tr>
<td>0.04 s⁻¹</td>
<td>0.28</td>
<td>0.22</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Steel B</th>
<th>1050°C</th>
<th>1150°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.01 s⁻¹</td>
<td>0.27</td>
<td>0.24</td>
</tr>
<tr>
<td>0.04 s⁻¹</td>
<td>0.34</td>
<td>0.28</td>
</tr>
</tbody>
</table>

**Table 2.** Peak strains (left) and peak stresses [MPa] (right) for the investigated conditions.

At each tested temperature and strain rate, the peak strains and stresses of steel B can be considered higher than steel A, being the differences between the average values higher than the standard deviation of the experimental data. The austenitic grain size before deformation was evaluated by metallographic technique. For both the materials, the average grain sizes were similar and about 400μm. Considering that the
chemical compositions of the steels are very similar except for niobium content, we can conclude that the observed differences between the mechanical results can be ascribed to Nb solute drag effects as the amount of Nb in solution is expected high under the selected testing conditions (both high reheating and deformation temperatures promote Nb dissolution and avoid occurrence of precipitation). Moreover, the entity of these differences is close to the values reported in the technical literature [7].

**Double compression tests**

The evolution of recrystallized fraction is shown for both steels A and B as a function of deformation temperature in Fig.4 and Fig.5, respectively for strain rates of 0.01s\(^{-1}\) and 0.04 s\(^{-1}\).

*Figure 4. Recrystallized fraction-strain rate 0.01 s\(^{-1}\).*

The recrystallized fractions obtained at strain rate equal to 0.01s\(^{-1}\) (Fig.4) showed different behaviours varying the test temperature: at 1050°C, the recrystallization kinetics of steel A is faster than that of steel B, whereas at 1150°C it was very similar. On the other hand, considering the highest strain rate (Fig.5), the effect of niobium in retarding the recrystallization kinetics is evident at both the investigated temperatures, being the recrystallized fractions of steel A (Nb-free) higher than those of steel B.

*Figure 5. Recrystallized fraction-strain rate 0.04 s\(^{-1}\).*

In Fig.4 it can be observed that, for the lower strain rate value (0.01 s\(^{-1}\)) and coarse initial grain size (about 400 μm), the recrystallization at 1150 °C deformation temperature of the C-Mn steel A has not been completed. This aspect was further investigated by means of metallography. Also the microstructure of Nb-microalloyed steel B deformed at 1150°C and 0.04 s\(^{-1}\) was examined in order to compare the recrystallization kinetics obtained by metallographic method with those by mechanic method (double compression tests). In Figures from 6 to 8, the cumulative distributions of the area covered by the grains and selected image post-processing results are reported for both steels.

*Figure 6. Steel A grains size cumulative distribution varying the inter-pass time.*

*Figure 7. Steel B grain size cumulative distribution varying the inter-pass time.*

*Figure 8. Steel A image analysis after 60s (a) and 130s (b) inter-pass time.*
Recrystallization is a thermally activated solid state reaction which occurs by nucleation and growth. The nucleation occurs predominantly at deformation heterogeneities and at the beginning of the transformation (site saturation condition) as can be seen in Figure 7. After the initial nucleation stage only growth occurs, i.e., grain boundaries migrate over the non-recrystallized strained matrix, lowering the total Gibbs energy of the system. The driving force is the stored energy in the material (i.e. crystalline defects) that decreases with time also due to concurrent recovery in the non-recrystallized regions. Therefore the strain heterogeneity and the occurrence of recovery may lead to a decrease in the speed of grain boundary migration during recrystallization and even to a recrystallization stagnation, as can be observed in Figure 4, 6 and 8 in fact state that for times longer than 60s (i.e. in the plateau) recrystallization does not proceed but only grain growth occurs.

The comparison of recrystallization kinetics obtained with double compression tests and metallographic observations is reported in Table 4 and 5.

<table>
<thead>
<tr>
<th>Steel B-0.04s⁻¹</th>
<th>X_{DC} [%]</th>
<th>X_{met} [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>inter-pass = 3s</td>
<td>45</td>
<td>23</td>
</tr>
<tr>
<td>inter-pass = 6s</td>
<td>70</td>
<td>32</td>
</tr>
<tr>
<td>inter-pass = 30s</td>
<td>100</td>
<td>45</td>
</tr>
</tbody>
</table>

Table 4. Comparison between mechanical (X_{DC}) and metallographic techniques (X_{met}) for recrystallization fraction of steel B.

<table>
<thead>
<tr>
<th>Steel A-0.01s⁻¹</th>
<th>X_{DC} [%]</th>
<th>X_{met} [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>inter-pass = 60s</td>
<td>65</td>
<td>20</td>
</tr>
<tr>
<td>inter-pass = 130s</td>
<td>80</td>
<td>/</td>
</tr>
</tbody>
</table>

Table 5. Comparison between mechanical (X_{DC}) and metallographic techniques (X_{met}) for recrystallization fraction of steel A. The value for inter-pass equal to 130s was not reported because grain growth already occurred as shown in Figure 8.

The data in Table 4, generally show a remarkable differences between the values obtained by the two methods: the mechanical approach seems to overestimate the recrystallized fraction and seems not to be sensitive to the grain growth, as observed for steel A. It shall be considered that the mechanical approach is influenced by different phenomena, such as the softening occurring in the non-recrystallized grains fraction and the influence of the strain rates on the stress values.

5. Conclusions

In this experimental work, the stress-strain curves and the recrystallization kinetics of Nb-alloyed and Nb-free steels were studied, by single and double compression tests at two test temperatures and strain rates. The recrystallization kinetics were also investigated by metallographic techniques on selected materials and thermo-mechanical conditions. It was identified the effect of niobium on the hot mechanical properties of steels and on the recrystallization kinetics during hot deformation. Moreover, a comparison was carried out between the recrystallization fraction obtained from the double compression test and by metallographic analysis at the same experimental conditions. The analysis resulted in remarkable differences between the values obtained by the two methods, in particular the mechanical approach appears to overestimate the recrystallized fraction.

References


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