

EFFECT OF NB IN SOLID SOLUTION ON THE AUSTENITE DECOMPOSITION KINETIC OF A V-Nb-Ti MICROALLOYED STEEL

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ABSTRACT

An experimental study was performed to assess the effect of Nb in solid solution on the austenite decomposition kinetic. The material analyzed was a V-Nb-Ti microalloyed steel with a total niobium content of 0.058 wt%. The experiments were carried out in a thermo-mechanical simulator Gleeble 3500. During the tests, the samples were reheated at 1170°C in order to dissolve all the Nb carbides, cooled down to 920°C, deformed in compression at this temperature, and finally cooled to room temperature at a fixed rate. The Nb precipitation kinetic at 920°C was measured using the stress relaxation technique. Different contents of Nb in solid solution prior to transformation were obtained by changing the holding time at temperature just after deformation. The phase transformation kinetic was derived from measurements of the electrical power needed to keep the sample cooling rate fixed. In these experiments, an important effect produced by the Nb in solid solution was observed: when reducing the holding time after deformation to decrease the amount of precipitated Nb, the polygonal ferrite reaction was strongly impaired, enhancing the formation of non-polygonal structures. The delay of the polygonal ferrite reaction was related to the segregation of Nb in solid solution to grain boundaries, where it may inhibit the ferrite nucleation or it may reduce the growth rate of ferrite by solute drag.

KEYWORDS

Phase Transformation, Microalloyed steel, Niobium Precipitation, Austenite Decomposition, Stress Relaxation.

INTRODUCTION

The niobium is commonly used as a microalloying element to increase the strength of the steel through different mechanisms. During hot rolling there is a strong decrease in the austenitic grain boundary mobility produced by Nb in solid solution [1] or forming carbonitrides [2-3]. Then, a reduction in the recrystallization rate occurs, enhancing the plastic strain accumulation. As the deformed austenitic grains are particularly effective in nucleating ferrite, an important microstructural refinement is finally produced. In addition, the Nb that remains in solid solution can precipitate in ferrite during cooling, increasing the strength due to particle dispersion hardening.

The microalloying additions may also play an important role in the kinetic of the austenite decomposition. It was shown that vanadium can produce grain refinement by promoting the formation of intragranular ferrite nucleated in non-dissolved precipitates boundaries [4]. It was also reported that Nb in solid solution can retard the austenite decomposition [5-7], with the subsequent grain refinement

and appearance of non-polygonal constituents. But the mechanisms associated with Nb in solid solution effect on the kinetic of the austenite to ferrite reaction is still a matter of controversy.

In this work an experimental study is presented to asses the effect of Nb in solid solution on the austenite decomposition kinetic. The paper is organized as follows: the experimental method is described in detail is Section 1, the results and some discussions are presented in Section 2, finally the conclusions are in Section 3.

1. EXPERIMENTAL PROCEDURE

In this investigation a low carbon V-Nb-Ti microalloyed steel with a total Nb content of 0.058 wt% was used (see Table I).

C	Mn	Si	V	Nb	Ti	N
0.11	1.23	0.23	0.048	0.058	0.017	0.005

Table I: Chemical composition in weight percent of the analyzed steel.

To study the niobium strain-induced precipitation effect on the austenite decomposition, thermo-mechanical tests were performed in a Gleeble 3500 machine. The extent of the strain induced Nb precipitation in austenite was controlled by changing the holding time after deformation. In this way, different amounts of the microalloying element were kept in solid solution previously to the phase transformation, which was monitored from measurements of the electrical power needed to keep the sample cooling rate fixed.

For the experiments, cylindrical samples of 10 mm diameter x 15 mm length were machined from the center of hot rolled plates. All the tests were conducted in a vacuum purged enclosed chamber under flowing high purity argon + 5% hydrogen to avoid decarburization and oxidation. The temperature was measured with a type K thermocouple spot-welded on the surface of the specimen, in its middle section.

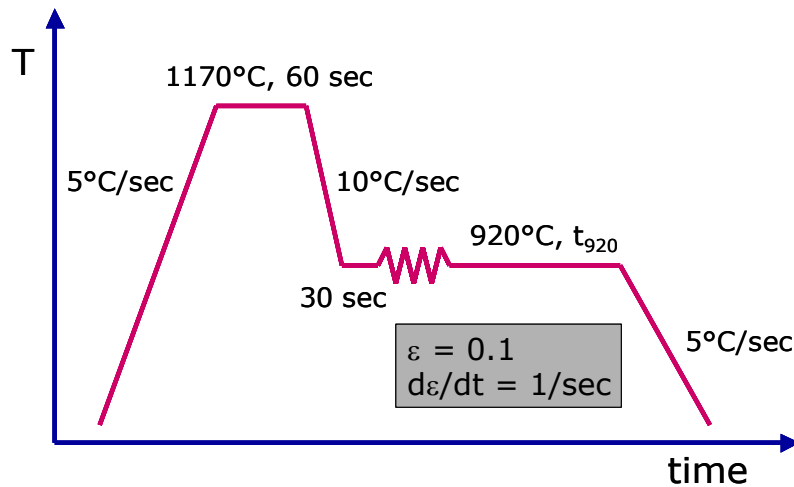


Figure 1: Schematic diagram of the thermo-mechanical tests performed at Gleeble 3500.

During the tests (see Figure 1), the samples were reheated at 5°C/sec up to the solutionizing temperature of 1170°C, held for 1 minute, and then cooled down at 10°C/sec to the deformation temperature of 920°C. Each sample was held at 920°C during 30 seconds to stabilize the temperature, and then deformed in compression to a true strain of 0.1 using a strain rate of 1/sec. After the deformation, the temperature and strain level were kept constant during a holding time (t_{920}) that ranged from 1 to 1800 seconds ($t_{920} = 1, 10, 30, 100, 300, 600, 900$ and 1800 sec). Then, in the last stage of the tests, the samples were cooled down to room temperature at 5°C/sec.

The objective of the reheating stage was to completely dissolve the niobium precipitates without having excessive grain growth. Due to the stoichiometric relation between the Ti and N additions (Table I), it was considered that both elements were fully precipitated as TiN, and neither of them was available for the formation of complex Nb carbonitrides. Then, the Nb precipitates were assumed to be NbC. The dissolution temperature of these carbides was estimated using the solubility product proposed by Siciliano and Jonas [8] that takes the effect of the Mn and Si additions into account; the obtained value was 1137°C. Due to the difference between the total dissolution and reheating temperatures, it was expected that almost all the Nb was dissolved during holding at 1170°C. The austenitic grain size resulting from the reheating cycle was measured on a sample quenched just before deformation, the result obtained was $d_\gamma = 60\text{-}70\text{ }\mu\text{m}$.

After compression, the strain-induced precipitation of Nb was followed using the stress relaxation technique [9]. From the changes in the slope of the force vs. logarithmic time curve the precipitation beginning and finish times were estimated.

Finally the specimens were cooled down to room temperature at 5°C/sec. In Gleeble machine, the sample thermal evolution may be controlled by applying an electrical current through the metallic specimen. Without current control, the sample natural cooling is a result of the amount of heat extracted by radiation and through the water cooled grips. In our experiment, due to the type of grips and the sample geometry, the natural cooling lead to a cooling rate of $\sim 10^\circ\text{C/sec}$ between 800°C and 600°C. Then, in order to get a fixed rate of 5°/sec, additional heating using controlled current was needed. The electrical power input was automatically determined by the thermal control system of the machine, and its time evolution was assessed in each test by measuring the sample voltage and the current supplied. During phase transformation, the heat released by the reaction reduces the amount of electrical power needed to keep the cooling rate fixed. Then, the analysis of the power vs. sample temperature curve was used to get information about the temperature range where the austenite decomposition took place. Some additional information regarding the type of reaction can also be inferred.

The samples were prepared for metallographic analysis using standard polishing techniques, etched in 2% nital, and examined by optical microscopy.

2. RESULTS AND DISCUSSION

In Figure 2 the stress relaxation curve obtained for $t_{920} = 1800$ sec is presented. The curve shows clearly two changes in the slope that were ascribed to the beginning (~ 25 sec) and end (~ 550 sec) of the NbC precipitation. These values were confirmed by comparison with the stress relaxation results for other holding times.

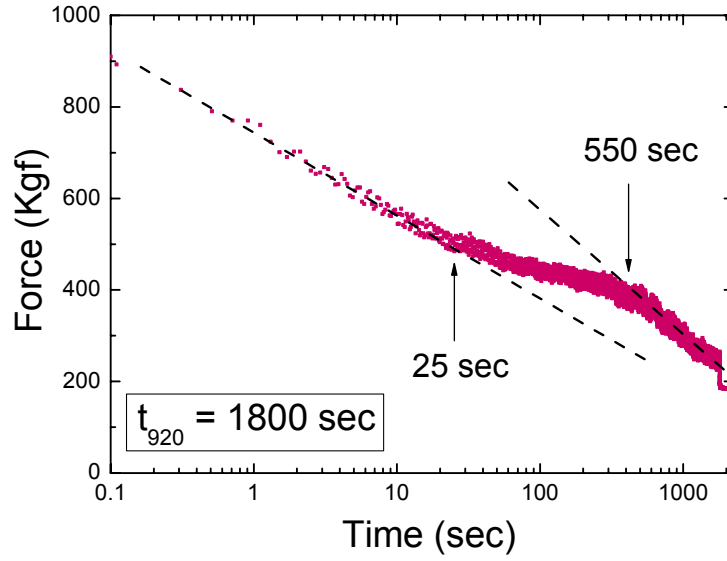


Figure 2: Stress relaxation curve measured at 920°C after a deformation of $\epsilon = 0.1$ using a strain rate of 1/sec. The times corresponding to the beginning and end of the NbC precipitation are indicated by arrows.

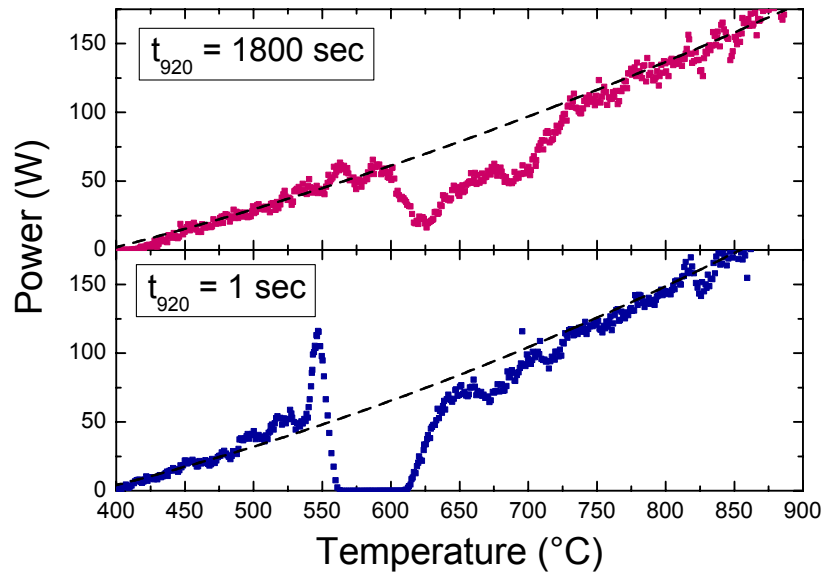


Figure 3: Electrical power needed to keep fixed the cooling rate at 5°C/sec during austenite decomposition. The samples were previously deformed at 920°C ($\epsilon = 0.1$ and $d\epsilon/dt = 1/\text{sec}$) and held at this temperature during 1800 and 1 sec.

The electrical power needed to keep the cooling rate fixed in the last stage of the test is shown for two holding times after deformation (1800 and 1 sec) in Figure 3. It can be seen that the electrical power

decreased slowly with temperature except in the region between 550°C and 750°C where sudden reductions in its value were needed to maintain the programmed cooling rate (5°C/sec). The inverse peaks in the power curve were associated to the occurrence of the austenite decomposition reaction. For $t_{920} = 1800$ sec two inverse peaks were observed at approximately 690°C and 625°C. On the other hand, only the low temperature inverse peak was clearly observed when $t_{920} = 1$ sec, and it was shifted to lower temperatures in comparison with the results obtained for longer holding times. It is important to notice that for $t_{920} = 1$ sec the electrical power went to zero in the temperature range between 560 and 610°C because of the released heat during the reaction. In spite of that, it was possible to keep a constant cooling rate of 5°C/sec in this test.

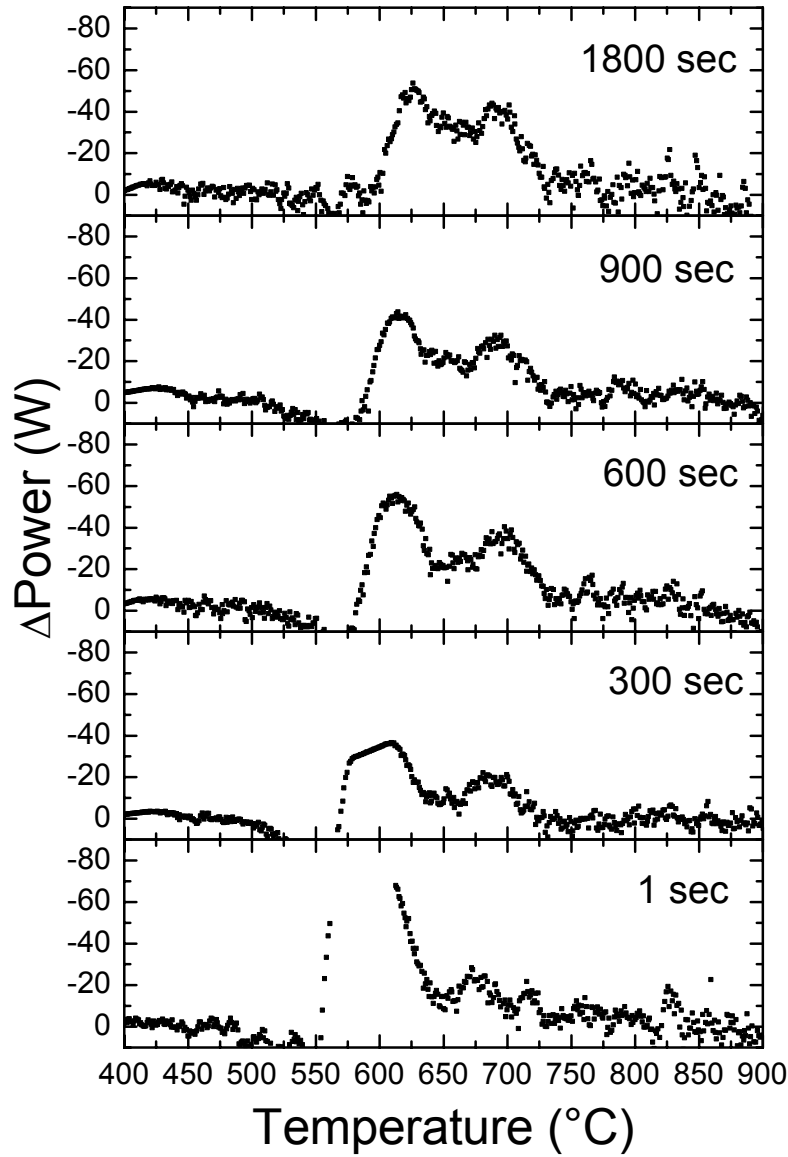


Figure 4: Power measurements after background subtraction. Results for samples previously deformed at 920°C ($\epsilon = 0.1$ and $d\epsilon/dt = 1/\text{sec}$) and held at this temperature during 1, 300, 600, 900 and 1800 sec.

The background in the power vs temperature measurements was estimated from a quadratic interpolation of the high and low temperature regions of each curve (dashed lines in Figure 3), then it was subtracted from the power data. The results obtained for $t_{920} = 1, 300, 600, 900$ and 1800 sec are presented in Figure 4. It is clear that as the holding time at 920°C was reduced, the high temperature peak continuously diminished in intensity and, at the same time, the low temperature peak grew and was shifted to lower temperatures.

These modifications in the type of transformation and in its temperature range were in agreement with the observed microstructures. In all the tests, the final microstructures were mixtures of polygonal ferrite (PF) and a constituent composed of non-polygonal ferrite + carbides (NPF + C). But the volume fraction of each constituent changed with holding time at 920°C . For the lower holding times, the structure was mostly composed of non-polygonal ferrite + carbides. Only small regions of polygonal ferrite in previous austenitic grain boundaries appeared in these samples (see micrograph for $t_{920} = 1$ sec in Figure 5). But, the polygonal ferrite volume fraction increased with holding time at 920°C , reaching a maximum value of approximately 50% for $t_{920} > 900$ sec (see micrograph for $t_{920} = 1800$ sec in Figure 5).

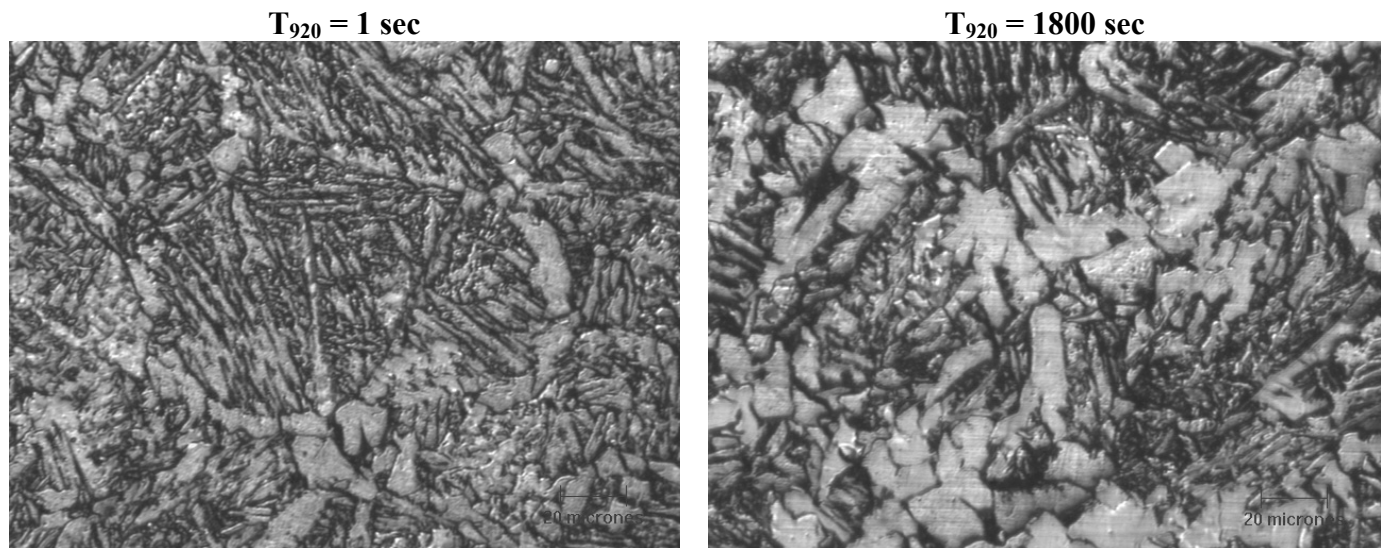


Figure 5: Optical micrographs of the samples held 1 and 1800 seconds after deformation and then cooled down to room temperature at $5^{\circ}\text{C}/\text{sec}$.

Taking the results obtained for the Nb precipitation kinetic into account (Figure 2), it is clear that as the Nb precipitation proceeded the formation of polygonal ferrite during the subsequent cooling was encouraged. Moreover, for the samples held at 920°C during times shorter than that corresponding to the onset of the Nb precipitation (~ 25 sec), the polygonal ferrite reaction was almost inhibited. On the other hand, when the precipitation at 920°C was finished ($t_{920} > 600$ sec), the polygonal ferrite volume fraction in the final structure increased to $\sim 50\%$. It is important to notice that, due to the low strain value ($\epsilon = 0.1$) and holding temperature (920°C), no recrystallization was expected to take place after deformation. Then, the changes in the final microstructure could not be related to differences in the austenitic grain size prior to phase transformation. On the other hand, it is clear from the stress relaxation curve (Figure 2) that recovery is proceeding. But the reduction in the dislocation density associated to recovery would produce the opposite effect than the one observed. Taking into account

that, as the precipitation proceeds, the amount of Nb in solid solution decreases, the behavior just mentioned may be explained considering that Nb in solid solution segregated to grain boundaries may inhibit the ferrite nucleation [7] or may reduce the growth rate of ferrite by solute drag [5-6]. Another explanation could be that the Nb precipitates can act as additional nucleation sites for the polygonal ferrite reaction. In order to study this possibility, partially transformed and quenched samples have to be analyzed in a transmission electron microscope, which is beyond the scope of this paper.

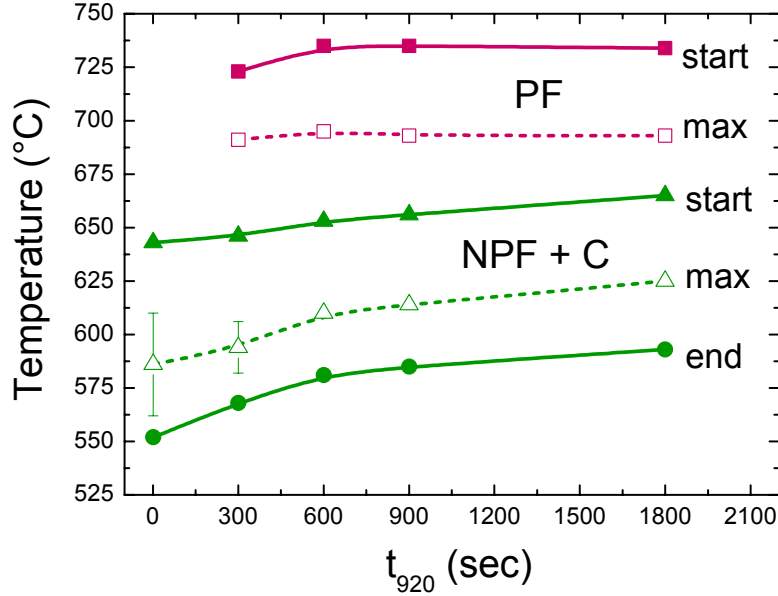


Figure 6: Critical temperatures corresponding to the beginning and end of the polygonal ferrite (PF) and non-polygonal ferrite + carbides (NPF + C) reactions.

In Figure 6 the temperatures corresponding to the beginning of the polygonal ferrite reaction (PF_{start}) and those corresponding to the beginning and end of the non-polygonal ferrite + carbide reaction ($NPF+C_{start}$ and $NPF+C_{end}$) are shown as a function of t_{920} . The temperatures associated to the maximum reaction rate of both transformation products (PF_{max} and $NPF+C_{max}$) are also plotted in this graph. All these temperatures were measured from the power curves once the background was subtracted. It is interesting to note that while the PF_{start} and PF_{max} temperatures were almost not affected by changes in the holding time, the NPF + C reaction was shifted to lower temperatures as the amount of Nb in solid solution increased (lower t_{920} values).

From the background subtracted power measurements, the peak intensities corresponding to both austenitic decomposition reactions (PF and NPF + C) were estimated using area integrals (inset in Figure 7). The polygonal ferrite peak intensity (I_{PF}) normalized by the sum of both peaks intensities ($I_{PF} + I_{NPF+C}$) is presented, as a function of the holding time after deformation, in Figure 7. Although the normalized PF peak intensity is not necessarily related in a quantitative way to the volume fraction of polygonal ferrite, there is a qualitative relation between both values. Higher normalized I_{PF} values corresponded to higher PF volume fractions. From the results presented in Figure 7 it is clear that at intermediate holding times after deformation (t_{920} values between 10 and 600 sec) there was a noticeable increase in the volume fraction of polygonal ferrite. On the other hand, for lower and higher

t_{920} values the PF volume fraction remained unchanged. These results confirmed the relationship between the holding time after deformation, which determined the extent of the Nb precipitation, and the increase in the polygonal ferrite volume fraction.

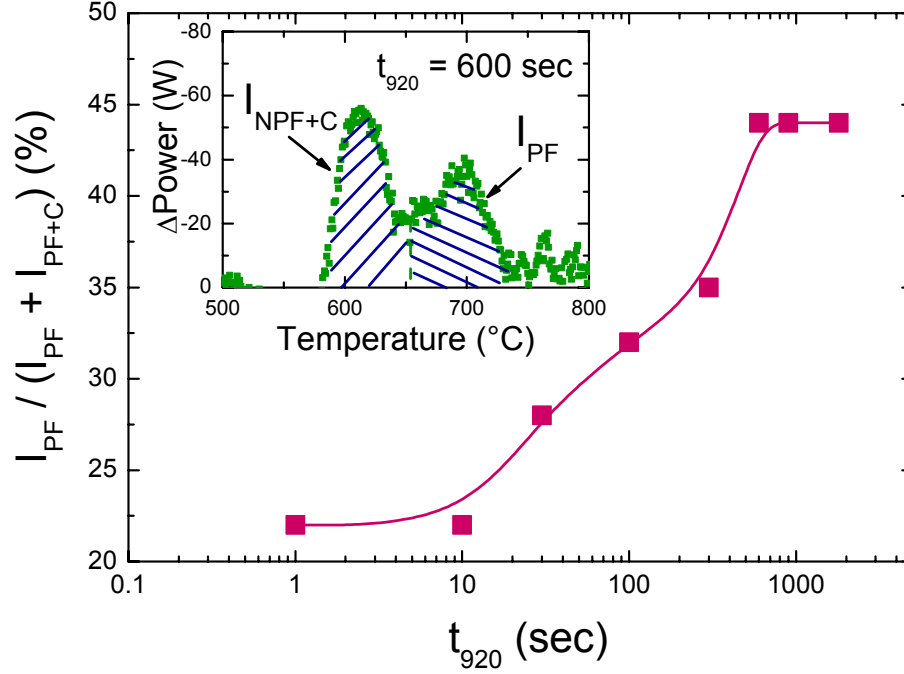


Figure 7: Normalized intensity of the polygonal ferrite peak ($I_{PF} / (I_{PF} + I_{NPF+C})$) as a function of the holding time at 920°C after deformation (t_{920}). Inset: determination of the PF and NPF + C peaks intensities (I_{PF} and I_{NPF+C}) from the background subtracted power curve.

There was also a relationship between the Nb precipitation and a decrease in the hardness of the samples (Figure 8). The drop in hardness was ascribed to the increment in the polygonal ferrite volume fraction. Regarding the Nb precipitation and its particle hardening effect, it is commonly accepted that Nb precipitates formed in austenite do not produce a noticeable hardness increase after austenite decomposition due to coarsening and the loss of coherency with the matrix. This is in agreement with our results that show no hardening associated to the Nb precipitation in austenite. The Nb that remained in solid solution after holding at 920°C was available for precipitation during or after phase transformation. But from measurements of Nb precipitation kinetic in ferrite [10-11], it was expected that the cooling rate was too high to allow the precipitation to proceed. Consequently, the net effect on hardness of the Nb precipitation in austenite was the softening produced by the increasing fraction of polygonal ferrite in a structure that otherwise was mainly composed of non-polygonal ferrite + carbides.

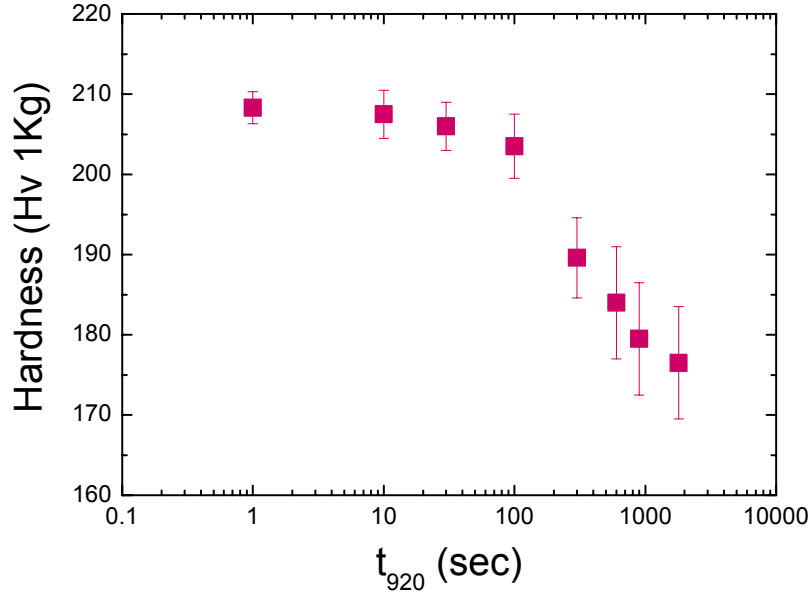


Figure 8: Final hardness as a function of the holding time at 920°C after deformation (t_{920}).

3. CONCLUSIONS

From the obtained results it is clear that the austenite decomposition is strongly affected by the extent of the Nb precipitation prior to the reaction. There is a direct relationship between the progress of the precipitation and an increase in the polygonal ferrite volume fraction in the final microstructure. Due to the low accumulated strain and holding temperature, no recrystallization was expected to take place after deformation. Then, the changes in the polygonal ferrite volume fraction could not be related to differences in the austenitic grain size prior to transformation. On the other hand, it was evident from the stress relaxation curves that recovery was proceeding. But the reduction in the dislocation density associated to recovery would produce the opposite effect than the one observed. The same is true regarding the possibility that some grain growth took place after precipitation. Taking into account that, as the precipitation proceeds, the amount of Nb in solid solution decreases, the behavior just mentioned may be explained considering that Nb in solid solution segregated to grain boundaries may inhibit the ferrite nucleation [7] or may reduce the growth rate of ferrite by solute drag [5-6]. Another explanation could be that the Nb precipitates can act as additional nucleation sites for the polygonal ferrite reaction.

It is interesting to note the different behaviors observed when analyzing the temperature ranges where the polygonal ferrite (PF) and the non-polygonal ferrite +carbides (NPF+C) reactions proceeded. While the PF start and finish temperatures were not affected by changes in the holding time after deformation, the NPF+C reaction was shifted to lower temperatures as the amount of Nb in solid solution increased. In order to understand this difference a detailed study, which is beyond the scope of this paper, is needed regarding the effect produced by Nb in solid solution on the nucleation and growth stages of both reactions.

There was also a direct relationship between the progress of the Nb precipitation in austenite and a decrease in the hardness of the samples that was ascribed to the increment in the polygonal ferrite volume fraction. This result was in agreement with previous published works [12] that suggest that Nb precipitation in austenite do not produce a noticeable hardness increase in the final microstructure.

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