

HIGH TEMPERATURE THERMOMECHANICAL PROCESSING OF NITROGEN BEARING Nb- ALLOYED STAINLESS STEEL

¹Andrei RUDSKOI, ^{1a}Georgii KODZHASPIROV, ²Jiri KLIBER

¹*Peter the Great St.Petersburg Polytechnic University, St.Petersburg, Russia,*
^agkodzhaspirov@gmail.com

²*VSB - Technical University of Ostrava, Ostrava, Czech Republic, EU*

<https://doi.org/10.37904/metal.2021.4097>

Abstract

The effect of High Temperature Thermomechanical Processing (HTMP) using hot rolling on the structure, mechanical properties of nitrogen bearing niobium alloyed 18Cr-10Ni austenitic stainless steel is presented. It has been found that the strengthening effect of HTMP depends significantly on the kinetics of deformation accumulation schedule. The change in rolling pressure with an increasing number of passes follow a similar pattern to the change in strength of the TMP treated rolling section. The basic factor determining the difference in structure formation of studying steels is thermodynamic stability of the carbide phase under the varying rolling accumulation schedules. TEM and light microscopy have been used in structural investigations. HTMP with a different accumulation schedule result in increasing of yield strength by 1.5 times compared to conventional heat treatment value.

Keywords: High Temperature Thermomechanical Processing (HTMP), Nb-alloyed austenitic stainless steel, dislocations, TEM, fragmentation, mechanical properties

1. INTRODUCTION

The austenitic stainless steels are widely used in industry because of their superior corrosion resistance and high (especially ductility and toughness) mechanical properties [1]. Relatively low yield strength (YS), however, is an obstacle to spread of this application. The typical strengthening methods of austenitic stainless steels include solid solution hardening and grain refinement [1-3]. It is known that Thermomechanical Processing (TMP) is one of the advanced strengthening resource saving technologies of metallic billets and parts of machine production [3-7]. As a result of TMP it is possible to increase strength and in the most cases it will not necessary to conduct heat treatment following by metal forming routinely. It has been reported that HTMP can give austenitic stainless steels grain refinement and substructure strengthening which rise strength without much reduction in ductility and toughness [3,6]. The effect of High-Temperature Thermomechanical Processing (HTMP) with one-time and fractional accumulation of deformation on the structure and mechanical properties of austenitic steel of type 18/10 stabilized by niobium is investigated.

2. EXPERIMENTAL

Steel of the following composition has been studied: 0.05 C; 18% Cr; 10% Ni; 0,05%N; 0.9% Nb (steel C-Cr-N-Ni-Nb). The specimens of the cross section 25 x 25 mm cut from forged ingots have been heated in an electric furnace at 1150 °C for 50 min, and then rolled after air cooling to the different deformation temperatures on the mill with roll's diameter 210 mm. Deformation has been realized with a various numbers of passes ($n = 1 \div 5$), followed by the accelerated water cooling. In the case of one-time deformation, the reduction (ϵ) was 10, 30 and 50%; in the case of fractional deformation, it was 10% in each pass. Deformation of 30 and 50% in each pass has been realized after air cooling preliminarily heated to 1150 °C samples, before the onset of rolling to the rolling temperature in the third

(1070 °C) and fifth (1020 °C) pass, respectively, in fractional deformation. The structure is investigated in cross sections parallel and perpendicular to the rolling axis. The mechanical test data are shown in **Table 1**. Metallographic investigation is undertaken on a light microscope; the dislocation's structure has been studied by TEM using a JEM-200 CX.

Table 1 Structural parameters and mechanical properties of C-Cr-Ni-Nb steel processed HTMP with different Numbers of passes (n) and Reductions (ϵ)

TMP parameters		Structural parameters					Mechanical properties			
n	ϵ (%)	D, (μm)	$\rho_0 \times 10^7$ (cm^{-3})	$\lambda \times 10^{10}$ (cm^{-2})	Δf (%)	Δr (%)	YS (MPa)	TS (MPa)	A (%)	RA (%)
1	10	81	6.4×10^7	2.5	15	0	320	626	53	72
1	30	51	8.7×10^7	1.0	50	5	362	634	31	66
1	50	40	16.1×10^7	1.5	70	25	279	631	51	64
3	30	47	10.2×10^7	1.5	80	0	331	664	49	65
5	50	35	22.1×10^7	2.5	95	0	388	687	43	66
CHT		85					253	610	58	69

Note: Conventional Heat Treatment - CHT (1100 °C, 50 min, quenching).

3. RESULTS AND DISCUSSION

The characteristic element of the microstructure in the initial (heating to 1150 °C for 50 min with subsequent quenching) state are relatively large grains $\sim 85 \mu\text{m}$, within which there are annealing twins and rounded inclusions (a few microns in size) uniformly scattered over the volume. Fractional deformation with increase in the total reduction, ϵ_{Σ} leads to qualitatively the same monotonic decrease in grain size, **D** and increase in the density of carbide-phase deposits, ρ_0 . There is pronounced extension of the grains in the direction of rolling. In contrast to fractional deformation, one-time deformation has a different influence on the structural parameters of steel. With increase in the reduction of one time deformation (one pass) to 50%, there is monotonic decrease in the grain size and increase in the density of carbide-phase precipitates, i.e. the variation in the structural parameters conforms to the same qualitative laws as in fractional deformation.

3.1 Structural and phase transformations in dislocations structure.

In the case of fractional deformation, increase in the total reduction leads to increase in the dislocation density. The greatest change is observed after the first pass: from 10^8 to $2.510^{10} \text{ cm}^{-2}$. Subsequent increase in has practically no influence on λ . After five passes, it remains at the level of a single 10% reduction. The spatial distribution of dislocations after the first pass is characterized by the presence of the volumes with a weakly expressed cellular structure, as well as volumes of fragmented substructure with the boundaries of fragments extended along the direction of rolling. Dislocations are short and winding, forming balls and knots. With fractional accumulation of the total reduction ϵ_{Σ} the fraction of the volume occupied by cellular structure decreases monotonically, while the fraction of the volume occupied by fragmented structure increases (**Table 1**) Thus, after the third pass, the fraction Δf of fragmented volume reaches 80; after fifth passes – 95%. With increase in ϵ_{Σ} , the degree of perfection of the fragmented structure increases, the quantity of broken boundaries decreases, and their dislocation structure is ordered. On average, the size of the fragments decreases, and the misorientation between them increases. The general law is increase in the extension of the fragments along the direction of rolling (**Figures 1a, b**).

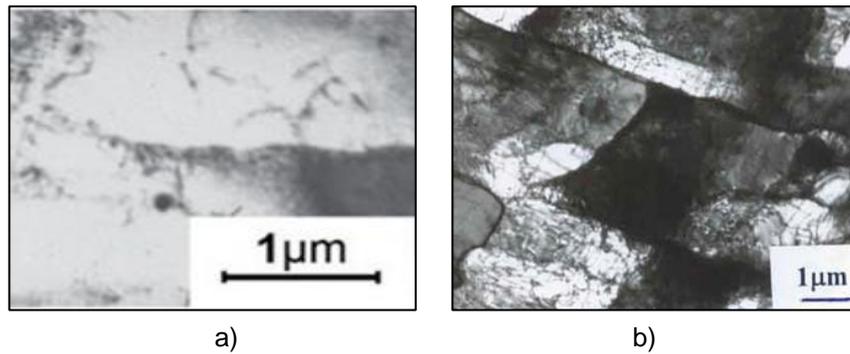


Figure 1 Microstructure (TEM) of steel C-Cr-Ni-Nb deformed in various conditions: a) extended fragments in steel subjected to deformation in one (a) and five (b) passes

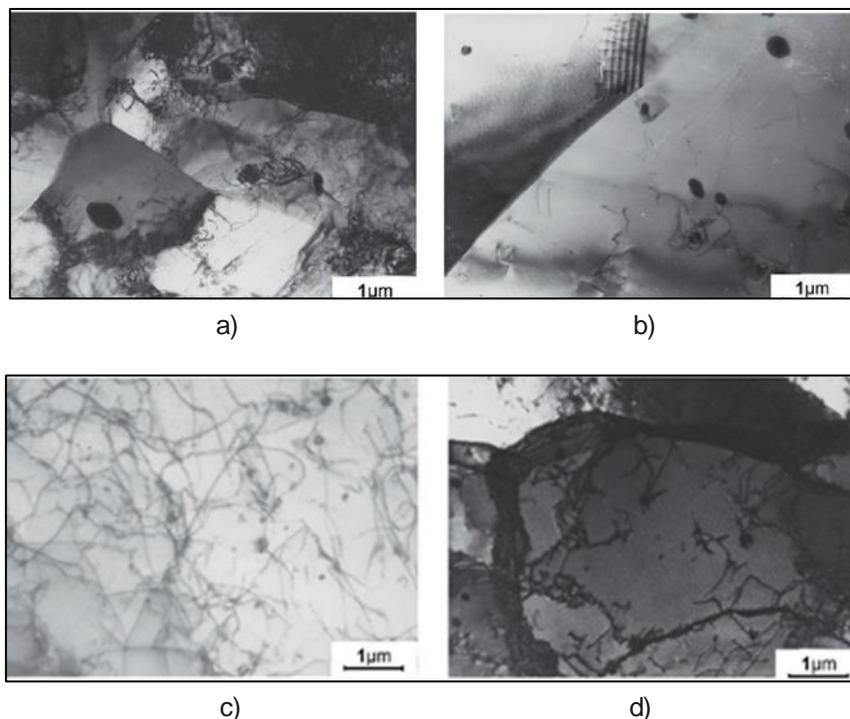


Figure 2 Dislocations structure (TEM) of steel C-Cr-Ni-Nb (a-d) subjected to 50% one time reduction: a) recrystallized sections against a background of strongly cold-hardened structure, b) dislocation free volume of recrystallized structure, c) uniformly distributed dislocations within recrystallized region, d) secondarily recrystallized region.

The formation of a dislocation network within the fragments (**Figure 1a**) is characteristic for studied steel. Volumes where fragmentation is observed against the background of a structure of deformational micro-twins are encountered (**Figure 1d**). With fractional accumulation of deformation, no signs of dynamic recrystallization are seen even after 50% reduction ($n = 5$). Structure formation in one time deformation also has distinctive features. The proportion of the volume occupied by cellular structure decreases monotonically with increase in ϵ . The fragmentation process with increase in ϵ , the proportion of the volume occupied by fragmented structure increases steadily. The most significant difference in the variation in structure in one-time deformation in comparison with the fractional case is that sections of dynamic recrystallization appear in the steel with increase in the one-time reduction. They are first observed after 30% reduction. Sections of recrystallized structure are encountered in two morphological modifications: a) non-dislocational ellipsoidal shape of the region, no greater than a few microns in size, with perfect high-angle boundaries; b) grains of size from 1 to 20 - 30 μm , usually of polygonal form, saturated by dislocations with different

density (**Figures 2 a-d**). Volumes of dynamic recrystallization appear after 30% one-time reduction. The proportion of the volume covered by the recrystallized structure, Δr is -5% when $\epsilon = 30\%$ and 25% when $\epsilon = 50\%$ (**Table 1**). If the development of the fine structure is traced within an individual recrystallization region, it is found that, in the course of continuing plastic deformation, the magnitude of which is measured from its moment of nucleation, this region passes through the same successive phases of fine-structure evolution as precede the onset of recrystallization: accumulation of dislocations up to densities corresponding to saturation, the formation of a cellular structure in the recrystallized grain only arises in the case where its dimensions exceed those of the cells characteristic for the given temperature-rate conditions of deformation of the steel, and fragmentation. The fragmentation is intensified and, if the deformation continues for long enough, new secondary regions of dynamic recrystallization may appear in the given primary recrystallized volumes. The process may then repeat itself again. The cyclical character of fine-structure evolution may easily be confirmed by observing and analyzing the types of dislocation structure corresponding to recrystallized grains of different size. In those which appear relatively late and hence are no greater than $0.2 \mu\text{m}$ in size, there are practically no dislocations (**Figure 2b**), although these regions are within strongly cold-hardened grains in a number of cases (**Figure 2a**). With increase in size of the recrystallized regions, the dislocation density within them increases (**Figure 2c**). Further increase in the recrystallized volumes leads to the appearance of a cellular structure, and to the onset of fragmentation (**Figure 2d**).

3.2 Features of carbide transformations.

The effect of alloying with Nb on the carbide transformations in steels of 18-10 type, including that associated with the formation of stacking faults on aging, was investigated in detail in [8,9]. Plastic deformation significantly influence the carbide transformations in this steel. The fractional accumulation of deformation leads to monotonic decrease in size of the carbide particles and simultaneous increase in their density (**Table 1**). In steel C-Cr-Ni-Nb, the dimensions D and volume density ρ_0 of the dispersed carbide particle (NbC) are practically unchanged in the course of deformation accumulation. In the case of one-time deformation an increase in the reduction degree is accompanied by increase in the density and size of the carbide precipitates. This is observed in both the fragmented and the recrystallized region, although more intensively in the latter case. To explain the influence of the method of strain accumulating on carbide transformations the following factors must be taken into account. The carbide NbC in reheating before the rolling (to $1150 \text{ }^\circ\text{C}$), is practically absent in steel. This is true, at least, for the dispersed fraction of NbC particles. The temperature conditions of rolling in the given experiment are chosen as a function of the accumulation procedure of deformation. With fractional deformation, the temperature of onset of rolling for all total reduction degrees is $1150 \text{ }^\circ\text{C}$. In one-time deformation, rolling with reduction $\epsilon = 10\%$ also begins at $1150 \text{ }^\circ\text{C}$, but rolling with $\epsilon = 30$ and 50% occurs after cooling samples preliminarily heated to $1150 \text{ }^\circ\text{C}$ down to temperatures corresponding to the third ($1070 \text{ }^\circ\text{C}$) and fifth ($1020 \text{ }^\circ\text{C}$) pass in fractional deformation [this is done so that the temperature at the end of deformation will be the same for the same total reduction degrees]. During deformation there is solution of finely disperse carbides result in of their interaction with the flux of moving dislocations. Freely moving carbon atoms enter the solid solution or segregate at dislocations in the form of Cottrell atmospheres. Increase in strain magnitude or rate is accompanied by increase in the degree of solution of the initial carbide phase, other conditions being equal. With subcooling of the steel or cooling in the pauses between rolling passes, the solid solution corresponding to the limit of solubility of C, or Nb atoms gradually becomes supersaturated, and breaks down, with the precipitation of carbide-phase particles. As more dislocations and inter-fragment boundaries are present in the metal volume, so the number of sites for carbide-particle precipitation increases and these particles are found to be more disperse and more homogeneously distributed over the volume. The kinetics of carbide transformations is constructed in resistance to these tendencies.

3.3 Mechanical properties

The laws of structural change observed in the present paper with one-time and fractional accumulation of deformation in steel 18-10 alloyed with Nb are of practical importance, since they permit the prediction of its behavior in rolling and in mechanical tests. For example, in the fractional accumulation of deformation,

monotonic increase in strength should be expected; this is associated with increase in the density of dislocations and deposits and also with increase in the proportion of cellular and fragmented structures. The degree of deformation at which a maximum of the strength properties is seen in steel subjected to one-time reduction coincides with the deformation ensuring a combination of high degrees of development of fragmentation and high carbide-phase density with the least development of recrystallization. Overall, the dependence of the yield strength on the magnitude and method of accumulation of the deformation may be schematically represented in the form in **Figure 3**.

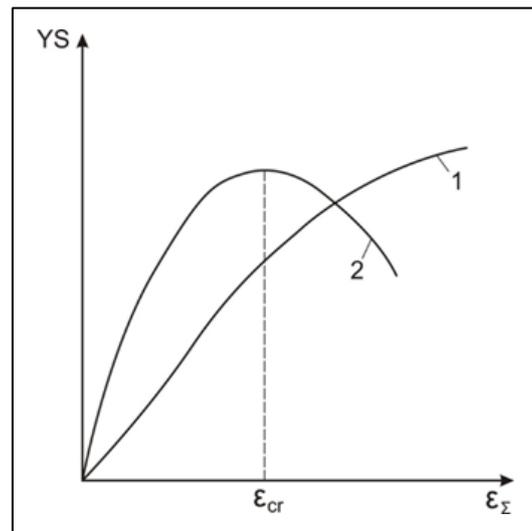


Figure 3 Schematic representation of the yield strength (YS) dependence on reduction (strain) magnitude and its strain accumulation schedule: 1) fractional, 2) one-time deformation.

An analogous correlation may be established between the structural changes and plastic characteristics of the steel.

4. CONCLUSIONS

- The HTMP promote a noticeable strength improvement of C-Cr-Ni-Nb steel.
- Monotonic growth in strength at the fractional accumulation of deformation result in increase of dislocations and precipitates density as well as increase of cellular and fragmented substructures quantities.
- Magnifying of one-time deformation reductions result in the dynamic recrystallization volumes appearance and more intensive dynamic recrystallization of Nb –bearing 18/10 type stainless steel and correspondingly the lowering of the strength.

REFERENCES

- [1] IRVINE, K.J.; LLEWELLYN, D.T.; PICKERING, F.B. *The Metallurgical Evolution of Stainless Steel*. Pickering, F.B., Ed. ASM, Materials Park: OH, 1979, p. 356.
- [2] OTTO A., PAULY T. The stainless steel industry's response to new challenges. In: *Proc. 6th European stainless steel conference*. Helsinki, 2008, p.31.
- [3] A.I. RUDSKOI, G. KODZHASPIROV, J. KLIBER, CH. APOSTOLOPOULOS. Thermomechanical Processing Of Steels And Alloys Physical Foundations, Resource Saving Technique And Modelling. *Materials Physics and Mechanics*. 2018, vol. 38, no. 1, pp. 16-25.
- [4] J.J. JONAS, M.R. BARNETT and P.D. HODGSON. Thermomechanical Processing. *Materials Processing Handbook*. Taylor & Francis; eds. J.R. Groza, J.F. Shackelford, E.J. Lavernia and M.T. Powers. CRC (Chemical Rubber Corp.) Handbook series, 2007, 29, p. 1.

- [5] A.J. DEARDO. New Challenges in the Thermomechanical Processing of HSLA Steels. *Materials Science Forum*. 2003, vols.426-432, pp. 49-56.
- [6] V.G. PROKOSHKINA, L.M. KAPUTKINA, YU. I.LOJNIKOV. Thermomechanical Treatment of Nitrogen-Containing Corrosion-Resistant Steels Various Structure Classes. *Materials Science Forum*. 2003, vols.426-432, pp.969-974.
- [7] X.J. LIANG, M.J. HUA, C.I. GARCIA and A.J. DEARDO. The Thermomechanical Control Processing of High Strength Steel Plate: A New View of Toughness Based in Modern Metallography. *Materials Science Forum*. 2013, vol. 762, pp.38-46.