Thermomechanical processing of Ti and Nb – alloyed stainless steels

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The effect of Thermomechanical Processing (TMP) using hot rolling on the structure, rolling pressure, mechanical properties of titanium and niobium alloyed 18Cr-10Ni austenitic stainless steels (type AISI 321) is presented. It has been found that the strengthening effect of TMP 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 different rolling accumulation schedules. TEM and light microscopy have been used in structural investigations. TMP with a different accumulation schedule result in increasing of yield strength by 1,3-1,7 times compared to conventional heat treatment value. The short-term high-temperature tensile tests showed that the higher room temperature strength level of TMP treated steel was also retained at high temperatures.

Keywords: Thermomechanical Processing (TMP) - Austenitic stainless steels - Dislocations - TEM - Fragmentation - Mechanical properties

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]. On a world basis, the austenitic share has long remained stable, at around 65% [2]. Relatively low yield strength, 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 [3,4]. 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 TMP can give austenitic stainless steels grain refinement and substructure hardening which rise strength without much reduction in ductility and toughness [3-5].
The effect of High-Temperature Thermomechanical Processing (HTMP) with one-time and fractional accumulation of deformation on the structure and mechanical properties of corrosion-resistant austenite steel of type 18-10 stabilized by titanium and niobium is investigated.

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EXPERIMENTAL

Steels of the following composition has been studied: 0.04% C; 18% Cr; 9% Ni; 02% Ti; (steel C-Cr-Ni-Ti)); and 0.04 C; 18% Cr; 9% Ni; 0.9% Nb (steel C-Cr-Ni-Nb). Samples of the cross section 25 x 25 mm cut from forged ingots are heated in an electric furnace at 1150 °C for 50 min, and then rolled after air cooling to the different deformation temperatures on a mill with roll's diameter 210 mm. Deformation has been realized with a various numbers of passes (n - 1÷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

TMP		Structural parameters					Mechanical properties			
n	ε, %	D,	ρ_ox10^7	λx10^{-10}	Δf	Δr,	YS	TS	A	RA
		mμ	cm^{-3}	cm^{-2}	%	%	MPa	MPa	%	%
C-Cr-Ni-Ti										
1	10	48	11,7 x10^7	1,5	10	0	313	654	53	71
1	30	24	20,7 x10^7	1,1	30	30	328	636	50	71
1	50	30	16,4 x10^7	0,4	10	90	246	618	55	64
3	30	39	14,0 x10^7	2,0	60	0	391	714	51	68
5	50	17	25,3 x10^{10}	2,3	90	0	410	681	46	70
CHT		50					266	579	61	76
C-Cr-Ni-Nb										
1	10	82	6,4 x10^7	2,5	15	0	320	626	53	71
1	30	51	8,7 x10^7	1,0	50	5	362	634	31	66
1	50	40	16,1 x10^7	1,5	70	25	279	631	51	64
3	30	47	10,2 x10^7	1,5	80	0	331	664	49	65
5	50	35	22,1 x10^7	2,5	95	0	388	687	43	66
CHT		85					253	610	58	69

Tab. 1 - Structural parameters & Mechanical properties of Steel C-Cr-Ni-Ti and C-Cr-Ni-Nb processed HTMP with different Numbers of passes (n) and Reductions (ε)

Tab. 1 – Parametri strutturali e proprietà meccaniche degli acciai C-Cr-Ni-Ti e C-Cr-Ni-Nb sottoposti a processo HTMP con diversi numeri di passaggi (n) e di riduzioni (ε)

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.

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

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 (Table 1; in steel C-Cr-Ni-Ti, the grain size D is - 50 mμ; in Ni-containing steel, it is - 85 mμ), 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 ρo in the steels. 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 steels C-Cr-Ni-Ti and C-Cr-Ni-Nb. In steel C-Cr-Ni-Ti, D and ρ_o vary nonmonotonically with increase in ε. When ε≤ 30%, D decreases, and ρ_o increases (Table 1). When ε=30%, the grain size reaches a minimum, and the deposit density a maximum. Increase in the reduction to 50% leads to increase in D and slight reduction in ρ_o.
In steel C-Cr-Ni-Nb, 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.
Structural and phase transformations in fine 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 1,5x·10^{10} in steel C-Cr-Ni-Ti and to 2,5x10^{10} cm^{-2} in steel C-Cr-Ni-Nb. Subsequent increase in ε_Σ has practically no influence on λ. After five passes, it is 2,3x10^{10} cm^{-2} in steel C-Cr-Ni-Ti; in steel C-Cr-Ni-Nb, 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. For steel C-Cr-Ni-Ti, long extended dislocations are typical. In steel C-Cr-Ni-Nb, they are short and winding, forming balls and knots. This indicates that in steel C-Cr-Ni-Ti the stacking fault energy is less than in steel C-Cr-Ni-Nb. Strongly split dislocations in steel C-Cr-Ni-Ti are shown in Fig. la. 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 Δ *f* of fragmented volume in steel C-Cr-Ni-Ti reaches -60%; after the fifth pass - 90%. In steel C-Cr-Ni-Nb the corresponding figures are ~80 and ~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 size of the fragments in steel C-Cr-Ni-Nb is larger, and the misorientation angles between the fragments are smaller, than in steel C-Cr-Ni-Ti. The general law is increase in the extension of the fragments along the direction of rolling (Fig. 1 b, c). The formation of a dislocation network within the fragments (Fig. 1 b, c) is characteristic (more for steel C-Cr-Ni-Nb). In steel C-Cr-Ni-Ti, volumes where fragmentation is observed against the background of a structure of deformational micro-twins are often encountered (Fig. 1d). In steel C-Cr-Ni-Nb, such volumes are less often encountered. With fractional accumulation of deformation, no signs of dynamic recrystallization are seen even after 50% reduction (n=5). This

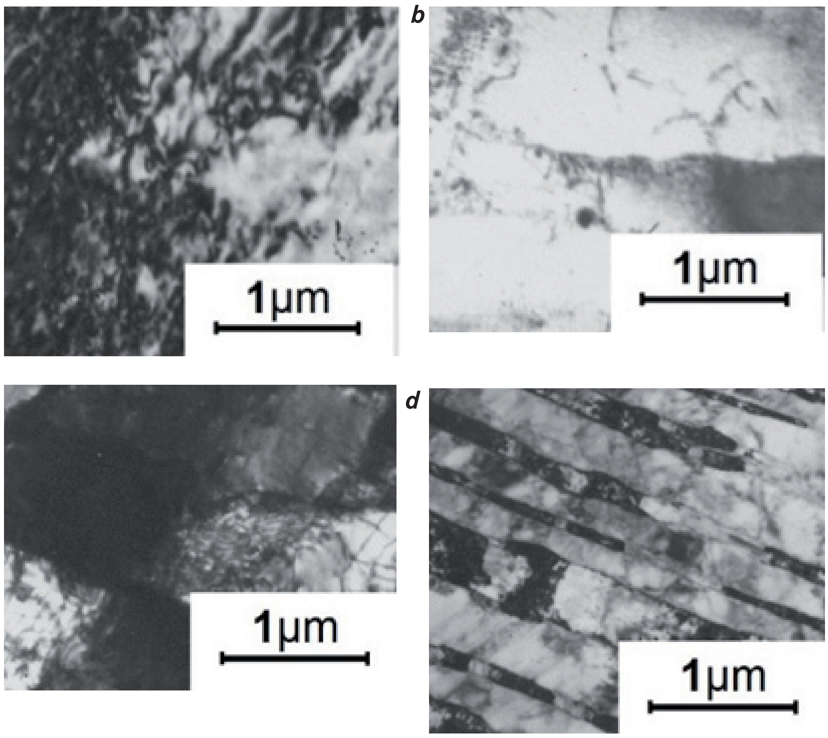

Fig. 1 - Fine structure (TEM) of steels C-Cr-Ni-Ti (a, b, d) and C-Cr-Ni-Nb (c) deformed in various conditions: a) strongly split dislocations in steel subjected to 10% deformation; b, c) extended fragments in steel subjected to deformation in one (b) and five (c) passes; d) fragmentation against a background of a microtwin structure in steel C-Cr-Ni-Ti.

Fig. 1 – Struttura fine (TEM) dell'acciaio C-Cr-Ni-Ti (a, b, d) e C-Cr-Ni-Nb (c) con diverse condizioni di deformazione: a) dislocazioni molto bloccate in acciaio sottoposto a deformazione al 10%; b, c) microstruttura frammentata estesa nell'acciaio sottoposto a deformazione con uno (b) e cinque (c) passaggi di laminazione; d) microstruttura frammentata su sfondo di una struttura microgerminata nell'acciaio C-Cr-Ni-Ti.

applies equally to both steels. Structure formation in one-time deformation also has distinctive features. In both steels, the proportion of the volume occupied by cellular structure decreases monotonically with increase in ε. However, the fragmentation process in steels C-Cr-Ni-Ti and C-Cr-Ni-Nb develops differently in characteristic parts: in steel C-Cr-Ni-Nb, with increase in ε, the proportion of the volume occupied by fragmented structure increases steadily, while in steel C-Cr-Ni-Ti the variation in Δf is non-monotonic (Table 1). Up to 30% reduction Δf increases; when $\varepsilon > 30\%$, Δf decreases. 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 (Fig. 2 a-d). Volumes of dynamic recrystallization appear after 30% one-time reduction in both steels, but with a different degree of recrystallization. The proportion of the volume covered by the recrystallized structure, Δr is -30% when $\varepsilon = 30\%$ and 90% when $\varepsilon = 50\%$ in steel C-Cr-Ni-Ti ; in steel C-Cr-Ni-Nb, the corresponding figures are -5% and -25% (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 [the 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 μm in size, there are practically no dislocations (Fig. 2c), although these regions are within strongly cold-hardened grains in a number of cases (Fig. 2a, b). With increase in size of the recrystallized regions, the dislocation density within them increases, and may reach the value λlim. (Fig. 2d).

Further increase in the recrystallized volumes leads to the appearance of a cellular structure, and to the onset of fragmentation (Fig. 2e). The lower stacking fault energy and the corresponding lower tendency of the dislocation structure of steel C-Cr-Ni-Ti to rearrangement means that, with accumulation of deformation in its structure, more elastic distortions accumulate than in steel C-Cr-Ni-Nb; consequently the dislocation structures there are more stressed and less stable. This leads to more intensive dynamic recrystallization in Ti - bearing steel.

Features of carbide transformations. The influence of alloying with Ti and Nb on the carbide transformations in steels of 18-10 type, including that associated with the

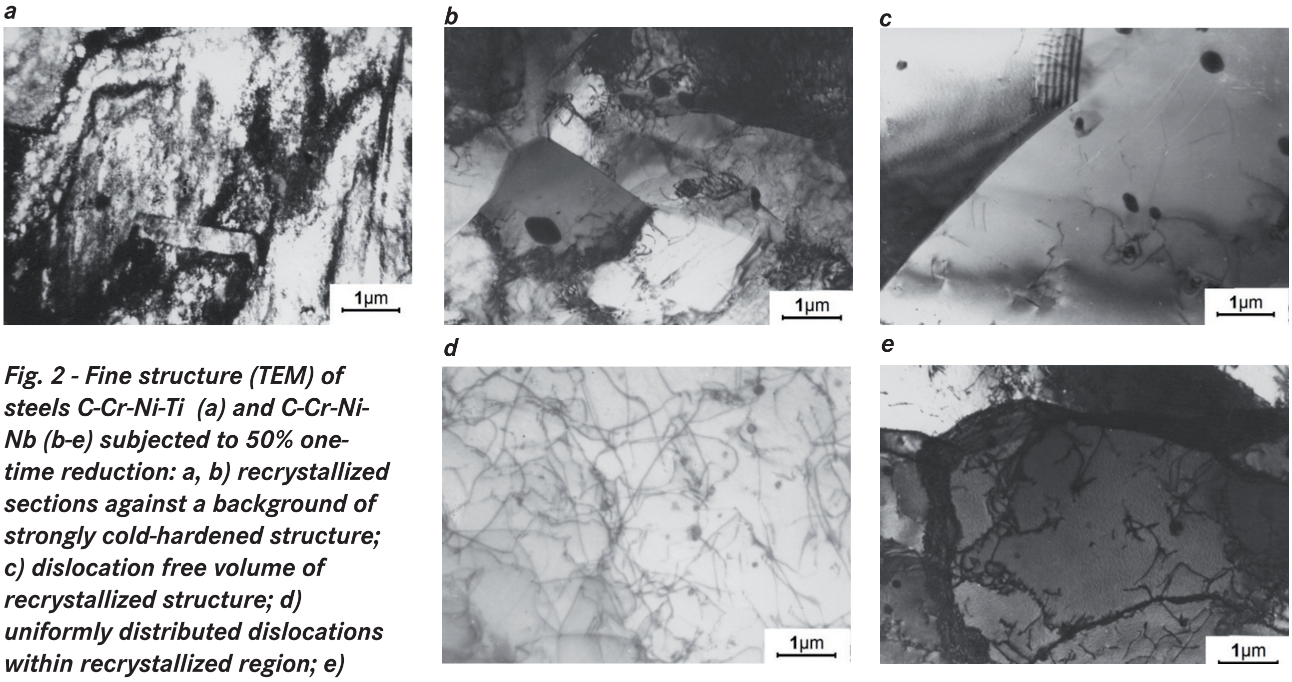

Fig. 2 - Fine structure (TEM) of steels C-Cr-Ni-Ti (a) and C-Cr-Ni-Nb (b-e) subjected to 50% one-time reduction: a, b) recrystallized sections against a background of strongly cold-hardened structure; c) dislocation free volume of recrystallized structure; d) uniformly distributed dislocations within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

Fig. 2 – Struttura fine (TEM) di acciai C-Cr-Ni-Ti (a) e C-Cr-Ni-Nb (b-e) sottoposti a riduzione del 50% in un passaggio: a, b) zone ricristallizzate nel contesto di una struttura sottoposta a forte incrudimento a freddo c) zona priva di dislocazioni nella struttura ricristallizzata; d) dislocazioni uniformemente distribuite entro la regione ricristallizzata; e) regione di ricristallizzazione secondaria.

formation of stacking faults on aging, was investigated in detail in [6,7]. Plastic deformation significantly influences the carbide transformations in these steels and introduces a series of significant differences in comparison with the results in. In steel C-Cr-Ni-Ti 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_o of the dispersed carbide particle (NbC) are practically unchanged in the course of deformation accumulation. These parameters remain at a lower level than in steel C-Cr-Ni-Ti for any ε.

In the case of one-time deformation in steel C-Cr-Ni-Ti the density of disperse carbide particles (TiC) decreases with increase in the reduction degree. With 50% reduction, it disappears, remaining only in volumes of recrystallized structure. In the one-time deformation of steel C-Cr-Ni-Nb the picture is different. 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 in the steels C-Cr-Ni-Ti and C-Cr-Ni-Nb the following factors must be taken into account.

1. The thermodynamic stability of the carbide phase in steels C-Cr-Ni-Ti (TiC) and C-Cr-Ni-Nb (NbC) is not the same. The carbide TiC is more stable, and its particles of different degrees of dispersity are present in steel C-Cr-Ni-Ti up to 1150°C. The carbide NbC is less stable and, in reheating before the rolling (to 1150 °C), is practically absent in steel C-Cr-Ni-Nb. This is true, at least, for the dispersed fraction of NbC particles.
2. 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 °C. In one-time deformation, rolling with reduction ε=10% also begins at 1150 °C, but rolling with ε=30 and 50% occurs after cooling samples preliminarily heated to 1150 °C down to temperatures corresponding to the third (1070 °C) and fifth (1020 °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].
3. During deformation in both steels, 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.
4. With subcooling of the steel or cooling in the pauses between rolling passes, the solid solution corresponding to the limit of solubility of C, Ti, 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

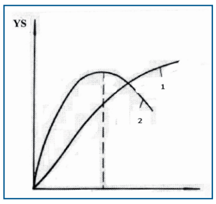

Fig. 3 - Schematic representation of the yield strength (YS) dependence on reduction (strain) magnitude and its strain accumulation schedule: 1) fractional; 2) one-time deformation.

Fig. 3 – Rappresentazione schematica della dipendenza del carico di snervamento (YS) dall'entità della riduzione e dalle modalità di accumulo della deformazione: 1) deformazione frazionata; 2) deformazione in una singola passata.

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.

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 Ti and 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 steels 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 point on the magnitude and method of accumulation of the deformation may be schematically represented in the form in Fig. 3. An analogous correlation may be established between the structural changes and plastic characteristics of the steel.

CONCLUSIONS

- The HTMP promote a noticeable strength improvement of C-Cr-Ni-Ti and C-Cr-Ni-Nb steels;
- 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 Ti - bearing steel in compare with Nb –bearing steel and correspondingly the lowering of the strength.

REFERENCES

1] Irvine, K.J.; Llewellyn, D.T.; Pickering, F.B. In 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. Proc. 6th European stainless steel conference. Helsinki, (2008), p.31.
3] G. Kodzhaspirov, I.Kim, E. Gulikhandanov, Proc.Int. Conf. on Thermomechanical Processing of steels, IOM, London, UK, (2000), p.392.
4] J.J. Jonas, M.R. Barnett and P.D. Hodgson: Thermomechanical Processing; Materials Processing Handbook, CRC (Chemical Rubber Corp.) Handbook series, Taylor & Francis; eds. J.R. Groza, J.F. Shackelford, E.J. Lavernia and M.T. Powers, 29 (2007), p. 1.
5] G.E. Kodjaspirov (Kodzhaspirov), L.P.Karjalainen, M.O.Speidel. Thermomechanical Strengthening of Nitrogen-Bearing Austenitic and Duplex Stainless Steels. Materials and Manufacturing Processes, Vol.19, Issue 1(2004) p. 87.
6] R. Honeycombc, J. Van Aswegen, and D. Warrington, "Role of packing defects in disperse-phase deposition," in: Structure and Mechanical-Properties of Metals [in Russian], Metallurgiya, Moscow, (1967), p. 177.
7] J. S. T. Van Aswegen and R. W. Honeycombc, "Segregation on precipitation in stacking faults," Acta Met, vol. 10, (1962).p. 262.

Trattamento termomeccanico di acciai inossidabili legati Ti e Nb

Keywords: Acciaio inossidabile - Processi termomeccanici

In questo lavoro vengono presentati gli effetti del processo termomeccanico (TMP), con laminazione a caldo, sulla struttura, sulla pressione di laminazione e sulle proprietà meccaniche degli acciai inossidabili austenitici 18Cr - 10Ni legati al titanio e niobio (tipo AISI 321). È stato riscontrato che l'effetto di aumento della resistenza meccanica dovuta al TMP dipende significativamente dalla cinetica delle modalità di accumulo della deformazione. La variazione della pressione di laminazione con un numero maggiore di passaggi segue uno schema simile alla variazione della resistenza della sezione di laminazione sottoposta a TMP. Il fattore fondamentale che determina la differenza nella formazione della struttura degli acciai in esame è la stabilità termodinamica della fase carburo nelle diverse modalità di accumulo della laminazione. Per gli studi strutturali sono stati utilizzati TEM e microscopia ottica. Nei processi termomeccanici con modalità di accumulo differente si è osservato un incremento del limite di snervamento da 1,3 a 1,7 volte rispetto al valore ottenuto con trattamento termico convenzionale. Le prove di trazione rapida ad alta temperatura hanno mostrato che il maggiore livello di resistenza meccanica a temperatura ambiente degli acciai trattati mediante TMP è stato mantenuto anche a temperature elevate.