HIGH TEMPERATURE THERMOMECHANICAL PROCESSING OF AUSTENITIC-FERRITIC STEEL

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Abstract

The effect of High-Temperature Thermomechanical Processing (HTMP) on the structure and mechanical properties of austenitic-ferritic steel has been studied. The dislocation structure observed in austenitic-ferritic stainless steels gave evidence that HTMP affected the strengthening and softening behaviour. Temperature - strain - time parameters are responsible for the substructure formation at the HTMP of duplex austenitic-ferritic steel. The effect of strain accumulation program in the formation of substructure and phase transformations as well and the relation between the changes in the crystal structure due to HTMP and the mechanical properties of the steels are considered.

Keywords: High-Temperature Thermomechanical Processing (HTMP), duplex austenitic-ferritic steel, dislocation structure, substructure, phase transformations

1. INTRODUCTION

A growing role in the economy of the state is played by the level of production of corrosion-resistant steels, successfully used in various industries of chemical, aviation, space and energy (including nuclear) industry. In the total volume of their production the largest proportion of Cr--Ni austenitic steel [1,2]. The wide application of such steels is due to their high chemical resistance in a wide temperature range, high plasticity and toughness, good weldability and high resistance to intergranular corrosion (IGC). However, their significant disadvantage, limiting the efficiency of use and scope, are low strength properties and especially the yield strength. The latter fact is compounded by the fact that one of the main tendencies in the world production of corrosion-resistant steels is the reduction of carbon content. Duplex corrosion-resistant austenitic steels differ from single-phase austenitic steels with higher yield strength, better weldability with good corrosion resistance in aggressive environments and increased resistance to intergranular corrosion and corrosion under stress corrosion cracking. Duplex steels are complex multicomponent alloys. In the process of metal working and heat treatment at different temperatures a variety of phase transformations takes place: a change in the quantity of δ-ferrite and austenite, accompanied by the redistribution of alloying elements between the phases, the decomposition of δ-ferrite with the formation of secondary austenite and σ-phase; the allocation of carbides and nitrides etc. The number of new grades of duplex corrosion-resistant steels is growing every year. The rapid development of nuclear power, increased safety requirements require the creation of reliable and compact nuclear power plant equipment with a sufficient margin of safety. In addition, it is necessary to take into account the aggravation of the issue of saving material and energy resources. In this regard, the use of resource-and energy-saving technology of TMP for strengthening of steels of this class seems to be quite relevant. By now a lot of research has been done on the effect of HTMP on the structure and properties of austenitic stainless steels [1-3], at the same time, there are very few data on the effect of HTMP on the structure, phase transformations and mechanical properties of duplex austenitic-ferritic steels [4].

2. EXPERIMENTAL

The experiments has been realized into the above mentioned hot deformation parameters during Duplex stainless steel 07CCr18Ni12TiV, the microstructure of which is shown in **Figure 1a**, has a good combination of mechanical properties (**Table 1**) in the original condition (water quenched from 1150 °C with holding for 2 h). **Figure 2** shows the change in the mechanical properties with aging at temperature up to 600 °C. The strength characteristics vary little with the aging temperature (T_{age}), increasing noticeably only with aging at 500 °C, so that no substantial increase in the yield strength occurs with this heat treatment.

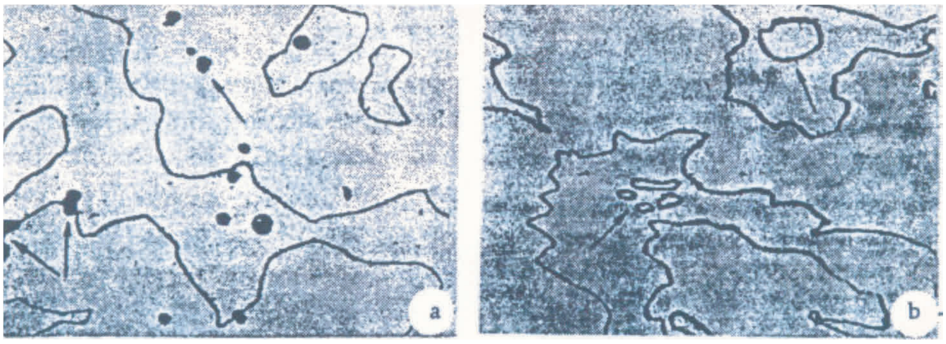
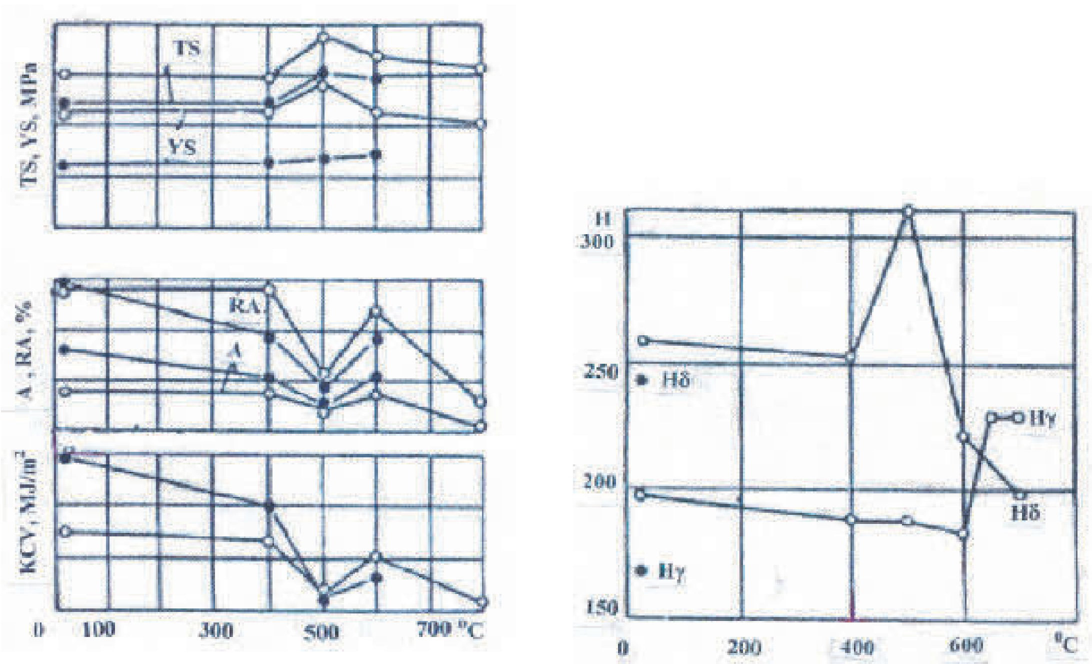


Figure 1 Microstructure of steel 07CCr18Ni12TiV . a) after quenching, Ti(C,N) carbonitrides indicated by arrows (x 160); b) after HTMP, γ' phase indicated by arrows (x 700)



a) b)

Figure 2 Mechanical properties (a) and microhardness (b) of steel 07CCr18Ni12TiV in relation to aging temperature after quenching (•) and HTMP (°)

Samples 22 x 22 mm in section from a forged bar of a commercial heat were subjected to HTMP: heating in an electric furnace to 1140 °C, rolling after cooling to 900 °C, in three passes, at a speed of 0.3 m/s, in a two-high rolling mill with 210 mm of rolls diameter (reduction ~ 40 %), followed by quenching in water. After HTMP and after quenching (original condition) some samples were aged at 400, 500, and 600 °C for 4 h, while after HTMP and after aging at 800 °C the sample were air-cooled.

The mechanical properties: yield strength (YS), tensile strength (TS), elongation (A), reduction in area (RA), impact strength (KSV) and the microhardness of δ ($H\delta$) and γ ($H\gamma$) phases were determined from the samples cut in the longitudinal direction relative to the rolling axis.

The chemical composition of the δ and γ phases and also large titanium carbonitrides was determined by means of the Cameca microprobe analyzer; a qualitative phase analysis of the carbonitrides was made by petrographic methods. Fracture surfaces were analyzed by scanning electron microscope (SEM). The fine structure of the steel was examined by transmission electron microscopy (TEM). The volume percentage of δ phase and also large carbonitrides was determined by quantitative metallographic analysis with the Quantimet instrument from an area ~10 mm^2 for each condition. The relative error in these measurements was 1 %.

3. RESULTS AND DISCUSSION

The results of mechanical tests showed, that HTMP leads to a considerable (185 MPa) increase of the YS of steel 07CCr18Ni12TiV and somewhat smaller increase (145 MPa) of the TS (**Table 1**). The elongation decreases ~40 %, while the reduction in area decreases slightly (**Table 1**). HTMP has different effects on the δ and γ components of the duplex steel. The increase in the microhardness of γ phase after HTMP is approximately double that of the δ phase. The fracture toughness decreases after HTMP, but remains fairly high (decreasing from ~2.7 to ~1.6 MJ/m^2).

Table 1 Mechanical properties and quantity of phases after quenching and HTMP

Treatment	YS (MPa)	TS (MPa)	A (%)	RA (%)	KCV (MJ/m^2)	H_γ	H_δ	Quantity (%)	
								δ phase	carbonitrides
Quenching	355	575	41.1	71.2	2.7	167	240	21.5	0.51
HTMP	540	720	25.4	65.5	1.6	197	257	19.8	0.28

The following qualitative changes are noted in the microstructure of the duplex steel after HTMP (**Figure 1**): secondary austenite (γ‘) is observed in ferrite grains, which points to decomposition of δ-ferrite during HTMP. The volume percentage changes correspondingly (see **Table 1**). Serrations appear on the δ/γ and δ/γ‘ interphase boundaries, which have often been observed after HTMP of austenitic steels [5].

Apart from the serration, the essential difference in the structure after HTMP and after standard quenching is the substantial reduction of the buck density of large (1-10 μm) titanium carbonitrides [Ti(C,N)], which evidently disappears in the process of high-temperature plastic deformation, although the deformation temperature is much lower than the temperature at which these carbonitrides go into solution (**Figure 1**, **Figure 3**). A qualitative phase analysis of these precipitates was made by petrographic methods. The ratio of the carbon and nitrogen concentrations was [C]:[N]$\approx$2:3. The chemical composition of the carbonitrides (**Table 2**) indicates that with their solution a substantial number of atoms of effective carbide-forming elements (titanium, vanadium) are transferred to the solid solution, which in the process of cooling again form special carbides or carbonitrides. In fact, transmission electron micrographs showed many finely dispersed carbides of the M7C3 type (density 10^{15} cm^{-3}) in δ-ferrite after HTMP, while almost none is observed in the original conditions (**Figure 3**).

Finely dispersed precipitates were not observed in austenite, in which the solubility of carbon is considerable higher, either in the original condition or after HTMP. A difference in the dislocation arrays is also observed in the δ and γ phases after HTMP. The dislocation density in austenite ($\rho\approx1.3\cdot10^{10}$ cm^{-2}) is approximately double that in ferrite. Parallel low-angle boundaries 0.2-1.0 μm apart are characteristic of austenite, while these boundaries are rarely observed in δ-ferrite formed during HTMP and generally have a large misorientation angle (15-20°).

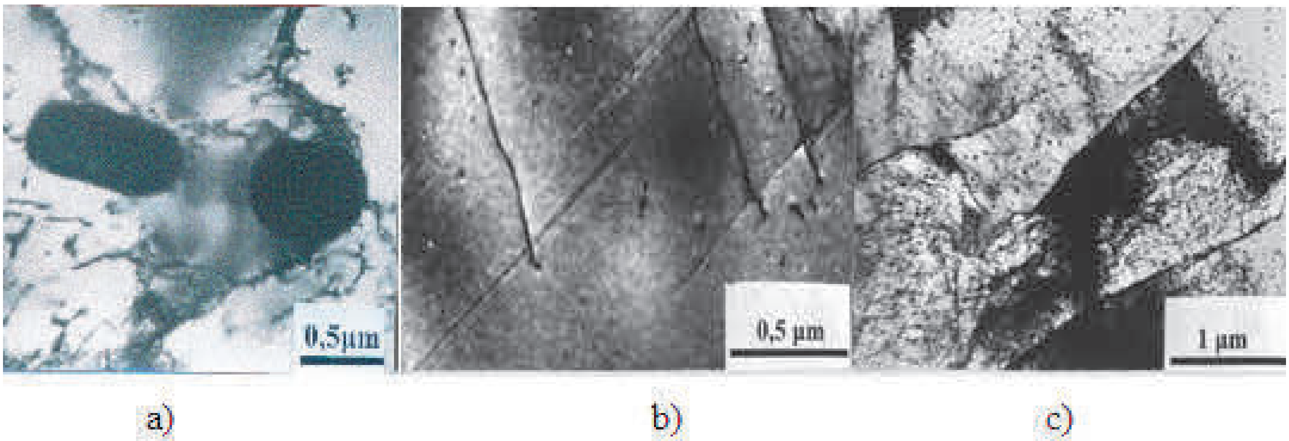


Figure 3 Fine structure of δ ferrite in steel 07CCr18Ni12TiV after quenching: a - MeC carbide; b- dislocation structure; c- fragmented substructure with finely dispersed precipitates after HTMP [own study]

According to the data, the chemical composition of δ and γ phases in steel 07CCr18Ni12TiV is different more and after HTMP (**Table 2**). The concentration of alloying elements in secondary austenite (γ') phase practically identical to the concentration in γ phase (after standard quenching from 1200 °C and aging the chemical composition of secondary austenite does not differ from that of δ ferrite).

Table 2 Composition of 07CCr18Ni12TiV steel after quenching and HTMP

Treatment	Phase	Composition (%)					
		Ti	V	Cr	Fe	Ni	(C+N)
HTMP	γ	0.9	1.0	17.5	66.8	14.5	-
	δ	0.9	1.5	23.2	65.0	8.7	-
	γ'	0.8	1.0	17.9	66.3	13.1	-
	Ti(C,N)	75.1	2.3	1.4	3.0	0.1	18.1
Quenching	γ	0.8	1.0	18.7	65.2	13.0	-
	δ	0.8	1.4	24.7	62.9	7.9	-
	Ti(C,N)	75.1	2.8	1.8	3.7	0.1	16.5

The variation of the strength and ductile characteristics and also the fracture toughness of steel 07CCr18Ni12TiV after HTMP and after quenching shows two ranges of maximum change in the mechanical properties, depending on the aging temperature (see **Figure 3**). One of them ($T_{age} \approx 500$ °C) is due to processes involving so-called 475 °C embrittlement, while the other (700-800 °C) is associated with precipitation of σ phase [6]. The mechanical properties change little with aging at temperatures up to 400 °C. The increase in strength the-aging at ~500 °C is due to hardening of δ ferrite, as can be seen in **Figure 3**. It is known that chromium laminates from the bcc solid solution in steels of similar composition at these temperatures, and decomposition of δ ferrite also occurs, with precipitation of dispersed intermetallic phase Ni_3Ti and Cr_2N nitrides. Aging at temperatures above 600 °C induce precipitation of carbides of the $M_{23}C_6$ type. The hardening of the γ phase associated with this cannot compensate the softening of δ phase, which is evident in the gradual reduction of the duplex steel (see **Figure 2**).

These changes in the structure and mechanical properties after aging are apparent also in the character of the fracture (**Figure 4**). Despite the substantially lower impact strength after HTMP, the fracture of the steel is ductile (dimpled) both after standard quenching and after HTMP (**Figure 4 a**). This type of fracture is also typical for samples aged at 400 °C. In the range of aging temperatures at which the ductility first declines the microphotography of the fracture surface changes abruptly. After standard quenching there are distinct traces nucleavage cracks (**Figure 4b**). After HTMP and aging at 500 °C the fracture is basically transcrystalline (**Figure 4c**), sections of intercrystalline fracture characterized by serrated grain boundaries are rarely observed (**Figure 4d**). These results indicate that HTMP of duplex austenitic-ferritic type is an effective method of increasing the strength with retention of fairly high ductility, toughness, and other operating characteristics. At the same time, the duplex material has several special characteristics associated with the difference in the processes of high-temperature deformation of bcc and fcc lattices, the presence of the δ/γ interphase boundary, the difference in the thermodynamic stability of solid solutions of δ and γ phases, etc.

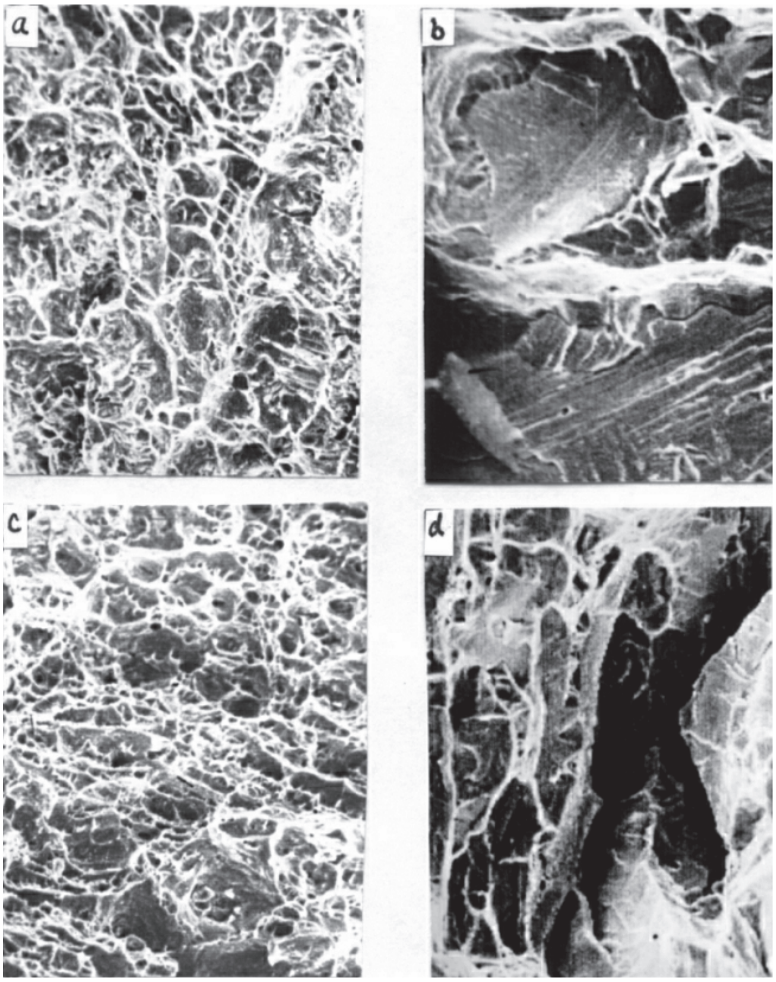


Figure 4 SEM fractographs of steel 07CCr18Ni12TiV (1000x). a) after quenching; b) after quenching + aging at 5000; c) HTMP + aging at 500 °C (typical section); d) HTMP + aging at 500 °C (section of intercrystalline fracture)

The HTMP stimulates decomposition of δ ferrite with precipitation of secondary austenite, and this must be taken into account if formation of γ' phase is undesirable. However, HTMP promotes more even distribution of carbide phase in the bulk of the steel, and therefore no negative effect of low-temperature quenching after plastic deformation is observed.

The principal hardening effect of HTMP is due to fragmentation of the bulk of the material in microscopic areas strongly misoriented with respect to each other (**Figure 3c**), in which the boundaries between then are effective barriers to plastic slip. The quicker occurrence of polygonization and annihilation of dislocations in δ ferrite as compared with austenite leads to less hardening of δ phase (**Figure 3**). An increase in the volume percentage of δ phase under these conditions of HTMP would evidently reduce the hardening effect. The serration of the δ/γ and δ/γ' interphase boundaries observed during HTMP inhibits formation and propagation of intercrystalline cracks. The high stability of the dislocation arrays in austenite increases the resistance of γ phase to aging in comparison with δ phase. While δ phase begins to soften during aging at temperatures >500 °C , austenite retains its strength up to much higher temperatures, and at 650-700 °C its strength even increases. Thus, the high resistance to softening at elevated aging temperatures of duplex steel subjected to HTMP is due to the stability of the fragmented austenitic component of the structure. An increase in the volume percentage of δ ferrite in the duplex steel subjected to HTMP should lead to a reduction of its heat resistance at aging temperature above 500 °C. The nonequilibrium of the supersaturated solid solution of δ phase creates the possibility of further strengthening of the duplex steel by aging. Aging at temperatures around 500 °C may additionally increase the strength of δ phase due to intermetallic and nitride precipitation hardening. The possibility of carbide hardening is not excluded if it is possible to fix atoms of C, Ti, and V (freed with the solution of primary carbonitrides) in the solid solution. On the whole, the effect of fragmentation and precipitation of finely dispersed precipitates on hardening is additive, and therefore the curves in **Figure 3** for steel 07CCr18Ni12TiV after quenching and after HTMP are identical in character. However, the fracture toughness is less sensitive to aging temperatures in the range of 20-600 °C after HTMP. Aging of steel 07CCr18Ni12TiV at temperatures up to 400 °C has no noticeable effect on its strength or ductile characteristics. However, aging at 500 °C, leading to a substantial increase of the yield strength and ultimate strength, cannot be recommended, since the ductility of the steel decreases.

4. CONCLUSION

- HTMP of duplex austenitic-ferritic type as for single-phase steels, is an effective method of increasing the strength with retention of fairly high ductility, toughness.
- The principal hardening effect of HTMP is due to fragmentation of the bulk of the material in microscopic areas strongly misoriented with respect to each other.
- Fracture toughness is less sensitive to aging temperatures in the range of 20-600 °C after HTMP

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