

## TENSILE TESTS ON STAINLESS STEELS IN TEMPERATURE RANGE 800 TO 1200 °C

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On different austenitic stainless steels tensile tests were carried out in the hot working temperature range for 800 to 1200 °C. The effects of  $\delta$  ferrite on recrystallisation, of  $\sigma$  phase on deformability and the effect of temperature on recovery, recrystallisation and grain growth were established.

*Key words:* austenitic stainless steel, tensile properties, high temperature, recrystallisation,  $\delta$  ferrite,  $\sigma$  phase

**Vlačna ispitivanja nehrđajućih čelika u temperaturnom intervalu 800-1200 °C.** Provedena su ispitivanja čvrstoće različitih austenitnih nehrđajućih čelika na povišenoj temperaturi 800-1200 °C. Utvrđeni su utjecaji  $\delta$  ferita na rekristalizaciju,  $\sigma$  faze na deformabilnost te temperature na oporavljanje, rekristalizaciju i porast zrna.

*Ključne riječi:* austenitni nehrđajući čelik, svojstva čvrstoće, povišena temperatura, rekristalizacija,  $\delta$  ferit,  $\sigma$  faza

### INTRODUCTION

In real conditions, austenitic stainless steels solidify in non equilibrium and a two phases microstructure of austenite and  $\delta$  ferrite is formed [1]. The aim of this work was to investigate the behaviour of both phases at tensile testing, especially to find out an eventual difference in the relaxation of the deformation energy and the effect of the two phases microstructure in workability. To achieve these goals, it was necessary to determine also the effect of annealing temperature and time on the share and shape of both phases in the microstructure. For experimental work the industrial steels AISI 316L, SS2343, X2CrNiMo 17-12-3 and X2CrNiMo 18-14-3 were used. These steels have by prescribed chemical composition a similar contents of base elements and f.i. all four steels have a content of chromium in the range of 16,5 % to 18 %, nickel of 10 % and 13 % in molybdenum between 2 % and 3 %.

### EXAMINATION

The chemical composition of the investigated steels is shown in table 1 and it is in all cases in the prescribed range. The test specimens were cut out from hot rolled plates. All steels have low carbon content and are without addition of elements strong binders of carbon and nitrogen, such as titanium and niobium. In the table are added also the chromium and nickel equivalents calculated according to the equations 1 and 2 in wt. %:

$$Cr_{eq.} = (Cr) + 2(Si) + 1,5(Mo) + 5(V) + 5,5(Al) + 1,75(Nb) + 1,5(Ti) + 0,75(W) \quad (1)$$

$$Ni_{eq.} = (Ni) + (Co) + 0,5(Mn) + 0,3(Cu) + 25(N) + 30(C) \quad (2)$$

proposed in references [2].

The tests were carried out in temperature range 800 °C to 1200 °C with the cross head speed of 5 mm/min. and computer recording of the force and the extension. The heating rate to the test temperature was of 600 °C/h and the holding time prior the tests was of 20 min. Specimens for metallographic examinations were cut out from the area of uniform elongation and the area of reduction of area of the tensile specimens. The examination was performed on an optical and a SE microscopes.

### EXAMINATION RESULTS

#### Tensile strength and flow curves

The recorded curves force  $F$  versus elongation  $\Delta L$  were transformed to true stress – strain dependences [3]. The true stress is given as quotient of the tensile force and the actual section  $A_i$  of the tensile specimen and the true strain  $\varepsilon$  is defined as the increment  $d\varepsilon = dL/L = -dA/A$ , with  $L$  the specimen length. If the plastic deformation does not involve the change of volume, the true strain is:  $\varepsilon = -\ln(A_i/A_0) = \ln(A_0/A_i)$ . The strain defined in this way is called logarithmic strain, also.

The tensile properties of the investigated steels in the tested temperature range are given in table 2.

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Table 1. Composition of the investigated steels in wt. %

Steel type	AISI	SS2343	X2CrNiMo	X2CrNiMo
	316L	–	17-12-3	18-14-3
	w/%	w/%	w/%	w/%
C	0,016	0,026	0,025	0,02
Si	0,48	0,36	0,33	0,34
Mn	1,49	1,51	1,6	1,53
P	0,030	0,033	0,039	0,032
S	0,002	0,025	0,002	0,002
Cr	16,6	16,87	16,99	17,85
Ni	10,03	10,74	10,62	12,83
Cu	0,32	0,4	0,34	0,38
Mo	2,06	2,53	2,51	2,83
Al	0,005	0,006	0,006	0,007
V	0,04	0,05	0,05	0,05
Ti	0,001	0,001	0,005	0,005
Nb	0,004	0,005	0,005	0,005
N	0,0239	0,0250	0,0478	0,0338
Cr <sub>eq.</sub>	20,89	21,69	21,71	23,04
Ni <sub>eq.</sub>	11,70	13,02	13,47	15,46
Cr <sub>eq.</sub> /Ni <sub>eq.</sub>	1,79	1,67	1,61	1,49

The comparison of the steels SS2343 in X2CrNiMo 17-12-3 with the steel AISI 316L shows that in temperature range 800 °C and 950 °C the strength is for about 20 N/mm<sup>2</sup> higher in the steel alloyed with sulphur and molybdenum. At higher temperature, from 1000 °C to 1200 °C, the tensile strength is virtually equal for all steels. The strength is higher at lower temperature range especially for the steel X2CrNiMo 18-14-3, which solidifies in austenite due to the ratio  $w.(Cr_{eq.})/w.(Ni_{eq.})$  lower than 1,5 [4]. The dependences force versus elongation is for the steels AISI 316L, SS2343 and X2CrNiMo 17-12-3 in table 2 are very similar. If the elongation at 1000 is compared, it is for the steel SS2343 ( $\Delta L \approx 47$  mm) smaller than that for the steels AISI 316L ( $\Delta L \approx 61$  mm) and X2CrNiMo 17-12-3 ( $\Delta L \approx 66$  mm). The elongation at 1100 °C was the greatest for the steels X2CrNiMo 17-12-3 ( $\Delta L \approx 86$  mm), SS2343 ( $\Delta L \approx 85$  mm) and AISI 316L ( $\Delta L \approx 71$  mm). The elongation is the smallest for all steels at the temperature of 1200 °C and indicates to a low workability by axial straining.

The flow curve, the dependence true stress  $\sigma$  versus logarithmic strain  $\varepsilon$  are for the steel AISI 316 shown for a different temperature in Figure 1. On these curves the area of homogenous axial deformation is situated in the initial part of the tensile specimen where the true stress is equal to the deformation resistance  $k_f$ . By tensile test, the force  $F$  achieves the maximal value  $F_{max}$  at the point of plastic instability, where the deformation becomes inhomogeneous and the engineering strength achieves the value  $F_{max}/A_0$ . In this part of the specimen the process of reduction of area is started and the plastic deformation is limited to a part of the specimen length and the deformation process is changed from axial to triaxial

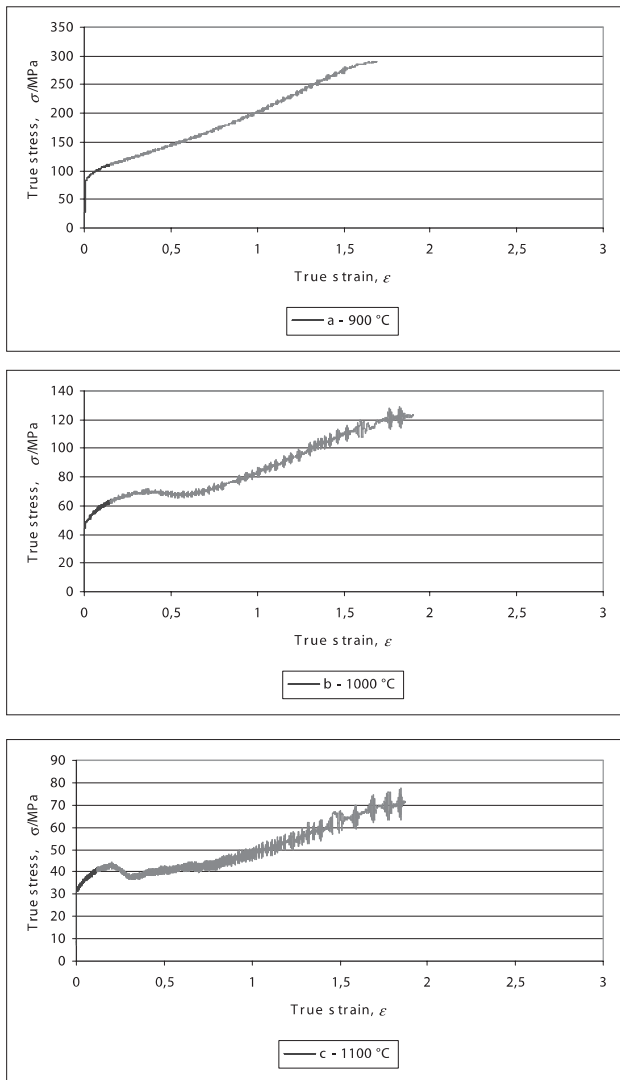
Table 2. Tensile properties of the steels in temperature range 800 °C to 1200 °C

Steel type	Temp.	Force	Tensile strength	Elong.	Reduction of area
	$T$	$F$	$R_m$	$A$	$Z$
	°C	kN	MPa	%	%
AISI	800	12,25	156	74	65
316L	850	10	127	88	96
	900	7,475	95	79	91
	950	5,35	69	63	60
	1000	4,25	55	99	94
	1100	2,8	36	117	90
	1200	1,8	23	74	81
SS2343	800	13,33	172	82	87
	850	10,78	139	109	94
	900	8,45	108	102	97
	950	6,25	73	101	98
	1000	4,75	56	75	88
	1100	3,05	36	137	91
	1200	2	23	67	73
X2CrNiMo	800	13,575	174	81	81
17-12-3	850	10,8	138	87	84
	900	8,425	108	100	96
	950	6,45	83	98	97
	1000	5	64	102	95
	1100	3,075	39	134	84
	1200	1,8	23	77	81
X2CrNiMo	800	14,9	191	81	86
18-14-3	850	13,225	170	58	85
	900	10,15	131	75	92
	950	5,9	77	113	96
	1000	4,85	62	91	81
	1100	3,3	43	96	69
	1200	2,05	26	73	67

type. In this range the true stress is slightly greater than the deformation resistance.

The flow stress increases to a determined extent of deformation and decreases afterwards. The increase is explained by strain hardening, while the lowering of the flow stress is explained partly because of the faster softening and partly with the formation of internal defects, first of all micropores in the striction area of the tested specimen. It is virtually impossible to evaluate the volume of micropores and their effect on the flow curves is not considered. For this reason, the values of flow stress can be used for the evaluation of the workability only until the maximal flow stress is achieved.

The testing rate of 5 mm/min is much smaller than the deformation rate at hot rolling. For this reason, the flow stress curves could be used only in a limited extent for the evaluation of the effect of temperature on the rolling workability.



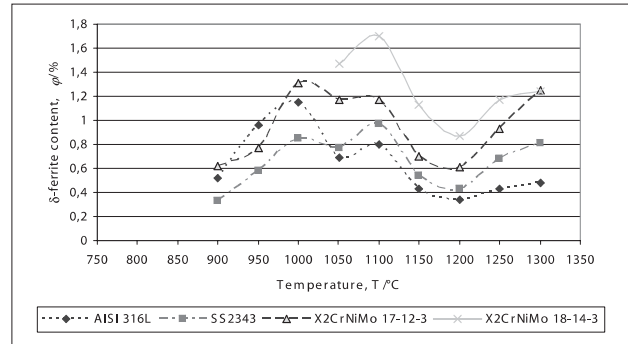
**Figure 1.** True stress in dependence of the logarithmic deformation for the steel AISI 316L and different test temperature (a - temperature 900 °C, b - temperature 1000 °C, c - temperature 1100 °C).

The true stress was by the same logarithmic strain greater by lower temperature and the dependence true stress versus true strain ( $\sigma-\varepsilon$ ) was modified with dynamical recovery and recrystallisation in the temperature range from 1000 °C to 1200 °C. For the steel AISI 316L in Figure 1b at 1000 °C and by the levels of true stress of  $\sigma = 70$  MPa and deformation of  $\varepsilon = 0,35$  the maximum is reached, than the true stress is diminished by with dynamic recrystallisation to  $\sigma = 65$  MPa by  $\varepsilon = 0,6$ . At higher temperature the maximal value of true stress is shifted to a lower strain.

### Microstructure, $\delta$ - ferrite and phase $\sigma$ before and after deformation

The microstructure of the investigated steels consists of austenite and  $\delta$  - ferrite and differs only slightly in the share and quantity of both phases. The solubility of different impurities in ferrite [5] diminishes the intensity of detrimental segregation on the boundary austenite -

ferrite. At higher temperature, the specific volume is greater and the temperature extension coefficient and the flow stress are smaller for  $\delta$  - ferrite. Ferrite is an unstable phase and it transforms to austenite with exchange of elements with diffusion between both phases with a rate depending on the temperature. The volume share of  $\delta$  - ferrite in dependence of the testing temperature is shown in Figure 2.

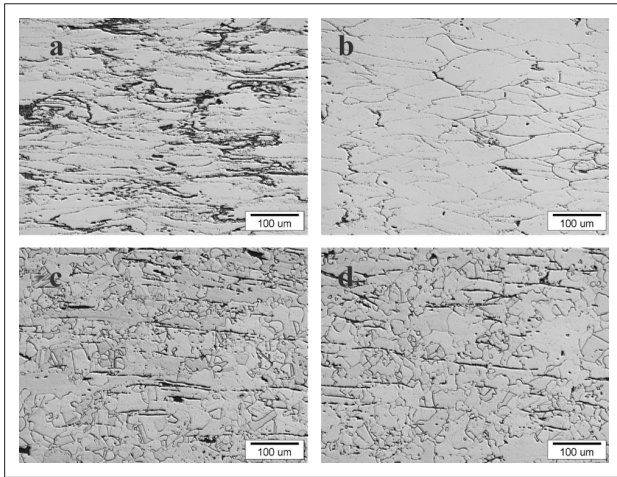


**Figure 2.** Content of  $\delta$  - ferrite in tested steels in dependence of the annealing temperature

The content of  $\delta$  - ferrite is the greatest after annealing at 1000 °C. At lower temperature the content of  $\delta$  - ferrite it is smaller because of the transformation to the non magnetic phase  $\sigma$ . In the steel X2CrNiMo 18-14-3 with higher contents of chromium and molybdenum the transformation  $\delta$  - ferrite to  $\sigma$  - phase occurs already at 1000 °C. The upper temperature for the formation of phase  $\sigma$  depends on the chemical composition of the steel and it can be shown in form of TTP (time - temperature - start) transformation curves [6]. The formation of  $\delta$  phase is faster at higher temperature.

$\delta$ -ferrite is found mostly at grain boundaries and its content does not change with the annealing temperature between 1000 °C and 1100 °C in the extent that could affect the deformation resistance and workability. On the contrary, the temperature affects strongly the kinetics of the change of shape of  $\delta$  - ferrite inserts. The driving force for this change is the great ratio of surface versus volume energy of ferrite inserts embedded in austenite. These inserts obtain a rounded shape or are divided in smaller segment of rounded shape.

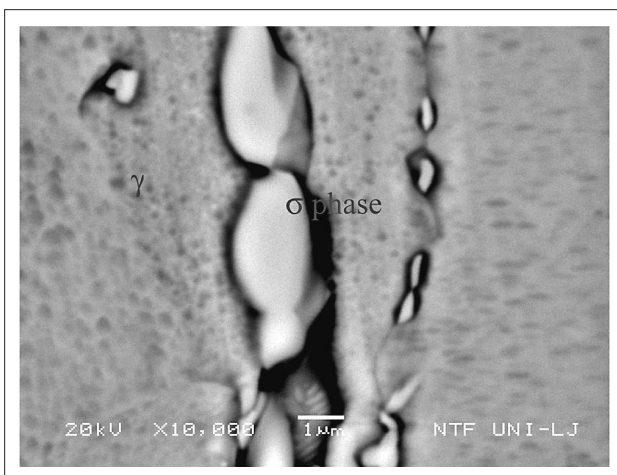
The shape of grains in the striction area near the fracture surface is at 800 °C and 850 °C strongly deformed and without marks of recrystallisation. Single small recrystallisation grains are found at the boundaries of  $\delta$ -ferrite and phase  $\sigma$  at 900 °C. The share of recrystallisation increases fast with the deformation temperature and at 1100 °C it amounts to more than 90 %, while at 1200 °C the recrystallisation is completed and the recrystallised grains are significantly coarsened. On Figure 3 the microstructure is shown for the striction area of the steel X2CrNiMo 17-12-3 tested at different temperature (800 °C and 1000 °C).



**Figure 3.** Microstructure in the striction area of fractured tensile specimens of steel X2CrNiMo 17-12-3 (a - 1 mm from the fracture, 800 °C; b - 10 mm from the fracture, 800 °C; c - 1 mm from the fracture, 1000 °C; d - 10 mm from the fracture, 1000 °C)

The plastic deformation affects strongly the kinetics of formation of phase  $\sigma$ . In the microstructure of specimens tested at a temperature below 1000 °C, in the middle of the layer of phase  $\sigma$ , a thin layer of ferrite with a lowered content of molybdenum was found. The formation of phase  $\sigma$  starts with nucleation at the phase boundary austenite – ferrite and grows on expense of the ferrite phase. By growing, the formed  $\sigma$  phase extracts chromium and molybdenum from ferrite. By higher annealing temperature also  $\sigma$  phase coalesces to rounded grains embedded in austenite. SE micrography of the phase  $\sigma$  in the steel X2CrNiMo17-12-3 are shown in Figure 4 for specimens tested at 900 °C. The content of molybdenum in phase  $\sigma$  was from 4 % to 8 % and that of chromium above 25 %, while the contents in the neighbour austenite were of 1 % to 2 % and 17 to 18 %.

In [6] the composition  $(\text{FeNi})_3(\text{Cr,Mo})_2$  is proposed for the phase  $\sigma$  in molybdenum stainless steels. The alloying elements chromium, molybdenum, tungsten, vanadium, silicon and niobium increase the stability of



**Figure 4.** SE micrography of different phases in the steel X2CrNiMo 17-12-3 tested at 900 °C

phase  $\sigma$  and enhance its formation. By adequate chemical composition of the steel,  $\sigma$  phase starts to grow on phase boundaries, on grain boundaries, on non coherent twin boundaries and at non metallic inclusions. Since, the phase  $\sigma$  extracts chromium and molybdenum, its formation causes the instability of carbide phases rich in both elements in the reaction area. Phase  $\sigma$  is hard and brittle and induces, for this reason, the fragility of stainless steels and decreases their corrosion resistance [6].

## DISCUSSION

In industrial condition, austenitic stainless steel are subjected to non equilibrium solidification which produces a two phases microstructure of austenite and  $\delta$  – ferrite. Depending on the content of base elements and other alloying elements as well as impurities, who may enhance the stability of  $\gamma$  – phase or  $\alpha$  – phase, the solidification is started with the primary formation of austenite or ferrite. The steels with the initial austenite solidification are more sensitive to solidification boundary segregation and hot cracking.

The results of this investigation and the quoted findings allow to explain the relation between deformation and the relaxation of the introduced energy, their connection to the composition of the steel and to the presence of  $\delta$ -ferrite in the deformed steel. However, data obtained with tensile tests do not allow to evaluate the workability of stainless steel at the initial hot rolling passes, especially the hot working behaviour of the steel with a solidification microstructure, termed initial steel workability.

Ferrite is in austenitic stainless steels the unstable phase and its content is diminished by keeping the steels at a temperature of sufficient diffusion rate of elements in substitutional solid solution. By the performed tests, the greatest content of ferrite was at the testing temperature of 1000 °C. However, also by higher testing temperature, the content of ferrite was not changed sufficiently to affect the steel deformability. Also the change of shape of ferrite inserts in the microstructure, mostly an initial phase of spheroidisation, which rate depends strongly on temperature, did not affect appreciably the deformability. The content of ferrite is diminished at low temperature because it transforms to the phase  $\sigma$ . In the steel X2CrNiMo 18-14-3 with higher contents of chromium and molybdenum the transformation of ferrite to phase  $\sigma$  is started already at the temperature of 1000 °C. The nucleation occurs mostly at the boundaries between both phases, austenite and ferrite, where the rate of migration of atoms to potential  $\sigma$  phase nucleation points is faster. In the process of growth, atoms of chromium and molybdenum diffuse from ferrite to the phase  $\sigma$ . At higher temperature also phase  $\sigma$  is spheroidised to isolate grains. In presence of phase  $\sigma$  the fragility of the stainless steel is increased. The rate of

formation of the phase  $\sigma$  is increased during the hot deformation and until the recrystallisation is ended.

In austenitic stainless steels molybdenum affects the static recrystallisation temperature and the rate of static recrystallisation and his effect is stronger by lower temperature. The interpass holding time for obtaining an equal share of recrystallisation is longer for steels with a higher content of molybdenum [7]. By the carried tests faster recrystallisation started at 1000 °C with nucleuses growing on grain and phase boundaries. For this reason, the recrystallisation rate is greater in presence of  $\delta$ -ferrite.

## CONCLUSION

The findings in this investigation and the quoted references justify the following conclusions:

The addition of the  $\alpha$  phase strengthening elements molybdenum increases the chromium equivalent and, by unchanged other alloying elements the quantity of  $\delta$ -ferrite formed at the solidification of the steel. In this way, the microstructure of the steel depends on the ratio of alloying elements increasing the stability of  $\alpha$ - and  $\gamma$ -phases. The quantity of  $\delta$ -ferrite is strongly increased by non equilibrium solidification of the steel and it is diminished at annealing of the steel before the hot working.

Molybdenum affects the ductility of austenitic stainless steels, decreases the workability, decreases also the rate of recrystallisation and increase the resistance to deformation by a temperature 1000 °C. At higher temperature the softening process is sufficiently fast to cover an eventual effect of molybdenum on the deformation resistance and deformability.

The tensile strength of the investigated steels in temperature range from 800 °C to 950 °C is for about 20 MPa higher for the steels SS2343 in X2CrNiMo 17-12-3

with higher contents of nickel, chromium and molybdenum, than that of the steel AISI 316L.

The greatest elongation was found for the AISI 316L, SS2343 and X2CrNiMo 17-12-3 at the temperature of 1100 °C. At the higher temperature of 1200 °C the elongation is very small for all tested steels. No explanation was found so far for this fast decrease of elongation.

In the investigated steels the content of  $\delta$  ferrite was the highest at 1000 °C. At higher temperature the quantity of ferrite was lower because it transformed to austenite with diffusion exchange of alloying elements. At lower temperature the content of ferrite is diminished by the transformation to the phase  $\sigma$ . In the steel X2CrNiMo 18-14-3 with the highest content of chromium and molybdenum, the transformation of  $\delta$  ferrite to the phase  $\sigma$  was observed already at the temperature of 1000 °C.

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**Note:** The responsible translator for English language is Franc Vodopivec, IMT- Ljubljana.