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# PROPERTIES OF STABILISED Ni-Cr CAST STEEL EXPOSED TO THE EFFECT OF CARBURISING ATMOSPHERE AND THERMAL FATIGUE

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*Preliminary Note - Prethodno priopćenje*

In this paper results investigation of an effect of niobium and/ or titanium as well as silicon on the resistance of 0,3%C-30%Ni-18%Cr cast steel to the carburising effect and thermal shocks at a temperature of 900 °C under the carburising atmosphere of carbon potential equal to 0,9% are shown. Eight test alloys were manufactured in which the content of niobium was changing in a range of 0-1,75%, that of titanium in a range of 0,03-1,00%, and of silicon in a range of 1,34-2,48% (wt. - %). Quantitative relationships were plotted to describe the effect of stabilizing elements on an increase of specimen weight and volume content of carbide phases in the external layers of specimens. Also the susceptibility to crack formation and mechanical properties after carburising process are shown. It has been proved that, with exception of the resistance to carburising effect, the examined elements deteriorate the cast steel properties.

***Key words:*** *Fe-Ni-Cr cast steel, carburising, thermal fatigue, surface cracks, carbides*

**Svojstva stabiliziranog Ni-Cr čeličnog lijeva izloženog utjecaju atmosfere za naugljičavanje i toplinskom zamoru.** U ovom radu prikazani su rezultati istraživanja utjecaja niobija i/ili titana te silicija na otpornost 0,3%C-30%Ni-18%Cr čeličnog lijeva u uvjetima naugljičenja i termalnih promjena kod temperature do 900 °C i potencijala ugljika do 0,9%. Izrađeno je osam legura kod kojih je sadržaj niobija od 0 do 1,75%, titana od 0,03 do 1,0% i silicija od 1,34 do 2,48% (mas. %). Prikazani su kvantitativni odnosi koji opisuju utjecaj stabilizirajućih elelmenata na porast mase uzorka i volumni sadržaj karbidnih faza u vanjskim slojevima uzoraka. Također je prikazana i osjetljivost na nastajanje pukotina kao i mehanička svojstva nakon procesa naugljičavanja. Utvrđeno je da, osim otpornosti na naugljičavanje, istraživani elementi pogoršavaju svojstva čeličnog lijeva.

***Ključne riječi:*** *Fe-Ni-Cr čelični lijev, naugljičavanje, termalni zamor, površinske pukotine, karbidi*

## INTRODUCTION

In austenitic cast steels, stabilizing elements are playing the two main functions [1, 2]:

- they reduce possible precipitation of chromium carbides, mainly along the grain boundaries, thus counteracting the impoverishment of matrix in chromium,
- they increase, through changes in the type and morphology of carbides, the creep resistance.

The elements used most commonly for this purpose are niobium and titanium. In the structure of alloys they are present in the form of carbides NbC and TiC. Added to the alloy jointly, they can also form complex carbides, e.g., (Nb, Ti)C [3].

In technical reference literature opinions are consistent as to the beneficial effect of niobium and/or titanium on an improvement of the creep resistance of austenitic cast steel under both oxidising and carburising atmospheres [3]. On the other hand, there are not enough data on an effect that these elements are supposed to exert on the thermal fatigue behaviour and other mechanical properties of this cast steel.

Therefore this study, using as an example 0,3%C-1,2%Si-30%Ni-18%Cr cast steel [4], makes an attempt at increasing the carburising resistance of nickel-chromium cast steel with its good resistance to thermal shocks well preserved, and this through extension of the chemical composition to include an addition of niobium and/or titanium and by raising the silicon content.

## TEST MATERIAL AND DESCRIPTION OF RESEARCH

When deciding about the content of niobium, titanium and silicon selected for this research, the intention was, on one hand, to keep it within the range investigated by other authors [1, 3, 5], while, on the other, it should produce the

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cast steel suitable for melting in furnaces with acid lining. As a result of the conducted research it has been decided that the content of the above mentioned elements will vary within the following range of values (wt. - %): 0,0 - 1,75 % Nb, 0,0 - 1,0 % Ti, 1,34 - 2,48 % Si. The content of the remaining elements was established at a level of the following values: C; 0,3 - 0,4 %, Mn; 0,8 - 1,0%, Cr; 18 - 20%, Ni; 29 - 32 %, P; 0,01 - 0,02 %, S; 0,01 - 0,015 %, rest - iron and the unavoidable impurities.

Eight test alloys were produce [6]. The compositions are given in Table 1. The specimens were prepared for the following examinations:

1. Weight increase due to carburising - specimens of dimensions 25×20×10 mm.
2. Thermal fatigue - ∅35×5 mm.
3. Mechanical tests: a) static tensile test (the shape and dimensions of specimens as well as the conditions under which the measurements have been conducted are described in [6]), b) V-notched impact test - 55×10×10 mm.

Table 1. **Chemical composition of the alloys tested / wt. %**
Tablica 1. **Kemijski sastav ispitivanih legura, mas. / %**

| Alloy | C | Si | Ni | Cr | Nb | Ti |
|---|---|---|---|---|---|---|
| 1 | 0,29 | 1,91 | 29,2 | 17,9 | 0,03 | 0,03 |
| 2 | 0,34 | 1,61 | 29,4 | 18,3 | 0,52 | 0,30 |
| 3 | 0,36 | 2,07 | 29,2 | 18,3 | 0,10 | 0,70 |
| 4 | 0,31 | 2,21 | 29,6 | 18,3 | 0,00 | 1,00 |
| 5 | 0,30 | 1,34 | 29,5 | 18,3 | 1,67 | 0,05 |
| 6 | 0,31 | 2,41 | 29,3 | 18,2 | 1,71 | 0,05 |
| 7 | 0,39 | 2,48 | 29,2 | 18,2 | 1,66 | 0,68 |
| 8 | 0,30 | 1,62 | 29,3 | 17,5 | 1,75 | 0,83 |
| Average content of other elements: Mn 0,91 - 0,97 %, P 0,013 - 0,019 %, S 0,009 - 0,012 % | | | | | | |

All specimens were carburised at a temperature of 900 ± 5 °C for 300 hours under the atmosphere of carbon potential equal to 0.9% [7]. Mean values of weight increment in specimens calculated in respect to the surface ($\Delta M/S$) are shown in Table 2. The confidence intervals were computed using Student-t test at a level of significance $\alpha = 0{,}05$.

Average volume fraction of carbides ($V_v$) in subsurface region of the carburised layer 100 $\mu$m wide was measured by means of Clemex 102 4C image analyser on polished cross-sections of gravimetric specimens (Table 2.). Examples of microstructures observed in the examined alloys are shown in Figure 1a, b. Specimens were etched with a solution of 3g $FeCl_3$, 10 ml HCl, 90 ml $C_2H_5OH$ for optical microscopy, and a solution of 10 ml $HNO_3$, 20 ml HCl, 30 ml glycerine at a temperature of 50 °C for outlining macro-structure. The results of structural examinations made on the steel as-cast, and after annealing and carburising are described in [6 - 8].

Table 2. **The results of measurements of the increment in specimens weight and the volume fraction of carbides due to the carburising effect**
Tablica 2. **Rezultati mjerenja prirasta mase uzoraka i volumnog udjela karbida uslijed naugljičavanja**

| Alloy | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 |
|---|---|---|---|---|---|---|---|---|
| $M/S$ mg/cm$^2$ | 11,46 ±2,06 | 8,80 ±0,85 | 9,37 ±0,90 | 8,72 ±0,64 | 7,97 ±0,81 | 8,04 ±0,75 | 6,18 ±0,45 | 7,65 ±0,59 |
| $V_v$ / % | 34,3 ±4,33 | 29,4 ±4,70 | 23,6 ±2,71 | 26,0 ±1,73 | 34,6 ±2,92 | 31,7 ±3,84 | 31,8 ±4,27 | 34,8 ±2,83 |
| Average from three measurements | | | | | | | | |

The thermal fatigue resistance of cast steel was evaluated computing the number of cracks formed in specimens due the shock preheating and cooling. The specimens were subjected to 30 cycles composed of :

- placing in furnace preheated to a temperature of 900 ± 5 °C,
- holding for 30 minutes,
- cooling in tank under running water.

Due to thermal fatigue, a network of cracks was formed on the specimen surface (Figure 1.c, 2.). To expose the

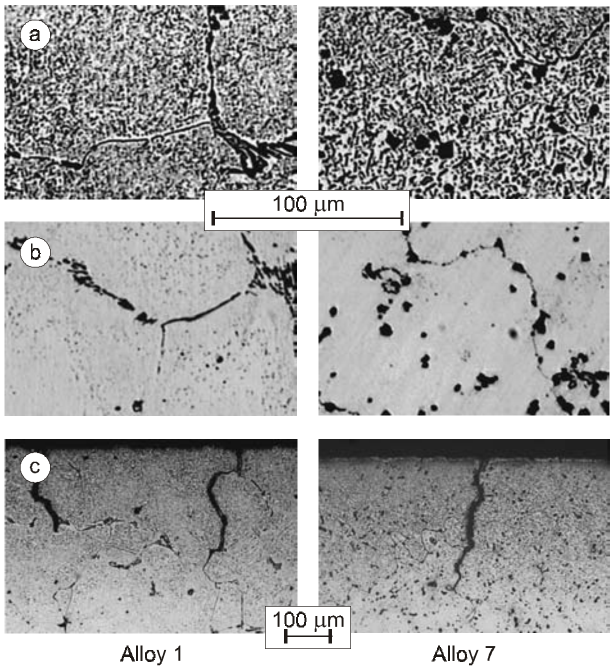


Figure 1. **Microstructure and thermal fatigue of alloys after the process of carburising: a) surface region, b) core, c) cracks visible against the background of carburised layer**
Slika 1. **Mikrostruktura i toplinski zamor legura nakon naugljičavanja: a) površinsko područje, b) jezgra, c) vidljive pukotine na pozadini naugljičenog sloja**

cracks completely, a penetrant made by Helling (Figure 2.a) was applied and photographs were taken. The photographs were next transformed into an 8-bit grey scale, were

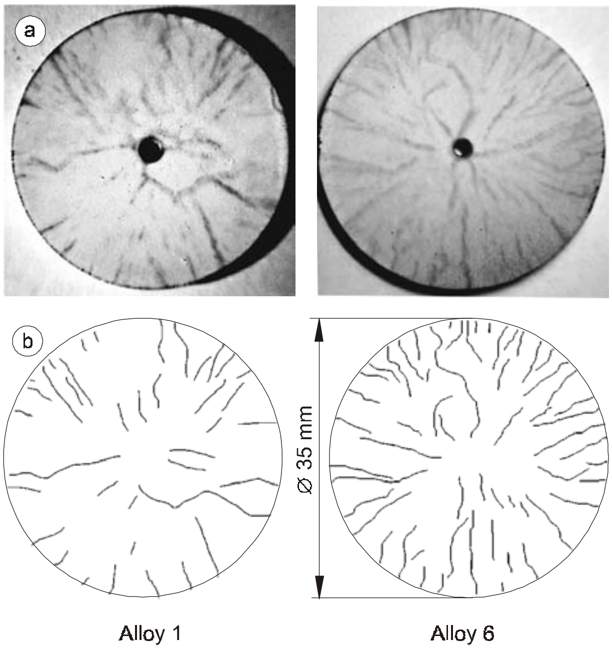


Figure 2. **Specimen surface after the process of shock thermal and cooling: a) after application of penetrants, b) binary image of cracks**

Slika 2. **Površina uzorka nakon toplinskog šoka hlađenja: a) nakon uporabe penetranata, b) binarni prikaz pukotina**

transported to a Visilog 4 programme, subjected to binarisation (Figure 2.b), and the percent content of cracks was computed (*P*) (Table 3.) [9].

Table 3. **The results of measurements of the content of cracks *P* / %**

Tablica 3. **Rezultati mjerenja sadržaja pukotina *P* / %**

| Alloy | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 |
|---|---|---|---|---|---|---|---|---|
| *P* / % | 2,6 ±0,07 | 3,2 ±0,08 | 3,2 ±0,10 | 3,9 ±0,14 | 2,7 ±0,09 | 2,6 ±0,11 | 4,2 ±0,08 | 3,7 ±0,09 |
| Average from three measurements | | | | | | | | |

The mechanical properties of cast steel were measured at temperatures of 20 and 900°C (Table 4.). The following properties were determined on the specimens: $R_{0.2}$ - 0,2 % proof stress, $R_m$ - tensile strength, $A_{10}$ - elongation, and impact resistance (KCV). The impact resistance was tested using an impact pendulum of 150J energy. Before placing the specimens on pendulum supports for impact test conducted at a temperature of 900 °C, the specimens were preheated at a temperature of 950 ± 5 °C for 30 minutes [9].

## RESULTS AND DISCUSSION

Due to carburising, in an external layer of the examined alloys, a carburised layer of different thickness, depending on the chemical composition, and with varied content, size and type of carbide phases was formed [7]; Figure 1.a. Also in the non-carburised core (Figure 1.b), the differences in the structure of the individual alloys are very notable [8]. A comparison of alloys microstructure leads to a conclusion that an increase in the content of niobium and/or titanium, obtained individually or jointly, affects in an important way the grain refinement and changes carbide morphology. The number of very fine secondary precipitates observed inside and around the grain boundaries decreases along with increasing content of the stabilising elements in cast steel. The large lamellar precipitates located mainly on the ternary grain boundaries disappear (alloy 1, Figure 1.b) to be replaced by large precipitates of a nodular shape (alloy 7, Figure 1.b).

Table 4. **The results of measurements of the mechanical properties of alloys**

Tablica 4. **Rezultati mjerenja mehaničkih svojstava legura**

| Alloy | $R_{0,2}$ / N/mm$^2$ | $R_m$ / N/mm$^2$ |
|---|---|---|
| | Test temperature: 20 / 900 °C | |
| 1 | 307,6 ± 12,9 / 87,1 ± 8,3 | 397,8 ± 10,7 / 141,0 ± 5,0 |
| 2 | 309,1 ± 14,3 / 82,3 ± 6,5 | 372,7 ± 12,6 / 134,7 ± 4,6 |
| 3 | 308,8 ± 14,0 / 80,0 ± 7,1 | 385,7 ± 12,7 / 128,7 ± 4,6 |
| 4 | 298,8 ± 15,8 / 82,4 ± 8,0 | 378,0 ± 12,2 / 125,8 ± 4,8 |
| 5 | 314,8 ± 12,7 / 95,0 ± 6,0 | 382,6 ± 14,3 / 140,1 ± 3,8 |
| 6 | 314,4 ± 12,1 / 83,8 ± 6,7 | 394,9 ± 16,3 / 134,2 ± 6,3 |
| 7 | 288,1 ± 14,4 / 75,2 ± 5,9 | 362,8 ± 11,4 / 117,1 ± 3,4 |
| 8 | 263,8 ± 13,7 / 81,6 ± 6,1 | 321,2 ± 11,1 / 125,6 ± 5,4 |
| Alloy | $A_{10}$ / % | *KCV** / J/cm$^2$ |
| 1 | 1,60 ± 0,1 / 14 ,8 ± 0, 6 | 27,0 ± 1,6 / 30,5 ± 3,0 |
| 2 | 1,80 ± 0,2 / 14,0 ± 0,6 | 25,7 ± 2,8 / 30,1 ± 3,2 |
| 3 | 2,20 ± 0,3 / 22,3 ± 0,7 | 27,2 ± 2,4 / 28,2 ± 2,8 |
| 4 | 1,90 ± 0,2 / 20,7 ± 0,5 | 26,3 ± 1,8 / 29,4 ± 3,4 |
| 5 | 1,60 ± 0,2 / 12,1 ± 0,5 | 24,2 ± 2,6 / 27,2 ± 2,6 |
| 6 | 1,80 ± 0,2 / 15,6 ± 0,6 | 23,4 ± 3,4 / 27,0 ± 2,4 |
| 7 | 2,10 ± 0,2 / 21,5 ± 0,7 | 28,8 ± 2,0 / 30,7 ± 1,8 |
| 8 | 1,90 ± 0,1 / 14,5 ± 0,6 | 23,5 ± 2,2 / 25,7 ± 3,1 |
| * Average from three measurements | | |

The equations of regression describing relationships between the chemical composition of cast steel (Table 1.) and the results of measurements (Tables 2. to 4.) were derived using a STATISTICA v.5.1 programmed. The equations together with their statistical parameters (coefficient of determination $R^2$ and statistics $F$) were presented and discussed below.

The increment in specimen weight due to carburising ($\Delta M/S$) was:

$$\Delta M/S = 9{,}22 - 4{,}39 \times C \times Nb + 0{,}06 \times Ti^{-1}$$

$$R^2 = 0{,}94,\ F = 36 \qquad (1)$$

The form of equation (1) indicates that under the conditions applied in these studies, the resistance to carburising increases along with increasing content of the stabilising elements and of carbon. The mechanisms by which niobium and titanium impede carbon diffusion inside cast steel are probably quite different. This results from the fact that niobium affects the value of *ΔM/S* in an approximately linear way, while the effect of titanium assumes a hyperbolic course, which is the reason why increasing the content of titanium in cast steel above 0,3 % is useless (Figure 3.a). Equation (1) also indicates that even a small change of carbon content in cast steel has a great impact on a resistance of this material to the carburising effect (Figure 3.b). On the other hand, the content of silicon in cast steel has no greater effect on the value of equation (1).

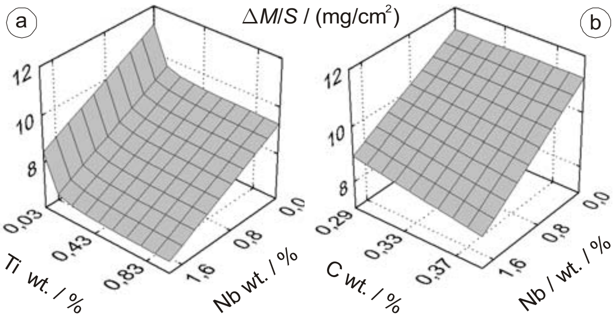


Figure 3. **Graphical illustration of the relationship shown by equation (1): a) for C = 0,35 %, b) for Ti = 0,03 %**
Slika 3. **Grafički prikaz odnosa prikazanog jednadžbom (1): a) za C = 0,35 %, b) za Ti = 0,03 %**

As a result of carburising, a carburised layer was formed on specimens, and it was characterised in respect of the non-carburised material by an elevated concentration of carbides (Figure 1.c, Table 2.). Generally it can be observed that along with increasing content of carbides in this layer the degradation of the mechanical and physical properties increases as well [1]. At the same time, the difference between the properties of the carburised layer and non-carburised core becomes more prominent. Both these processes are said to affect adversely the properties on the entire cast wall cross-section. An immediate effect is, for example, the reduced resistance of cast steel to thermal fatigue, or deteriorated plastic properties [2, 9].

The volume fraction of carbides ($V_v$) in subsurface region of the carburised layer, related to the content of the examined elements, is expressed by the following equation:

$$V_v = 20,1 + 5.8 \times Nb \times Ti + 10.5 \times Si^{-1} + 0,3 \times Ti^{-1}$$
$$R^2 = 0,92,\ F = 15 \qquad (2)$$

Examining the form of equation (2) it is relatively easy to trace a physical relationship between the value of $V_v$ and the content of niobium and silicon in cast steel. Increasing niobium content causes binding of larger volumes of carbon in carbides, thus leaving respectively more of chromium in the matrix. This, in turn, promotes the precipitation of a higher amount of chromium carbides during carburising. As a result, a dangerous increase in the content of carbide phases in a carburised layer, accompanying an increase in niobium content, is observed (Figure 4.a). On the other hand,

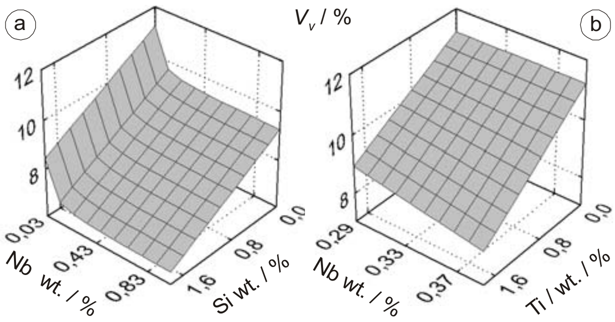


Figure 4. **Graphical illustration of the relationship shown by equation (2): a) for Ti = 0,03 %, b) for Si = 2,0 %**
Slika 4. **Grafički prikaz odnosa prikazanog jednadžbom (2): a) za Ti = 0,03 %, b) za Si = 2,0 %**

quite a beneficial effect has increasing of silicon content in cast steel, mainly due to the fact that it reduces the content of carbides in a unit volume of the carburised layer (Figure 4.a). A single addition of titanium to cast steel is also said to have a beneficial effect, though - according to equation (1) - adding this element in an amount above 0,4 % is useless (Figure 4.b). Because of the assumptions adopted previously, it is not recommended to introduce to cast steel niobium together with titanium (Figure 4.b).

Irrespective of the reasons which account for this varied effect of the examined elements on the values of equations (1) and (2), from an analysis of the relevant factors it follows that under the conditions adopted in these studies, a maximum cast steel resistance to the carburising effect (and at the same time such that would not impair other cast steel properties and cause changes in the structure of a subsurface carburised layer) is achieved with the following chemical composition (wt. %): 0,39 % C, 2,48 % Si and 0,4 % Ti.

The number of cracks (*P*) formed in cast steel due to thermal fatigue is described by the following equation:

$$P = 2,53 - 1,45 \times Nb + 1,28 \times Ti + 4,95 \times C \times Nb$$
$$R^2 = 0,95,\ F = 27 \qquad (3)$$

The source of the observed cracks are mainly structural microstresses formed due to the alternate preheating and cooling of specimens. They result from very great differences in physical properties (mainly in the values of the coefficients of thermal expansion [10]) of the matrix (austenite) and carbides present in the structure (NbC, TiC, $M_{23}C_6$). From the form of equation (3) it follows that the number of cracks depends, first of all, on the content of titanium, and next on that of niobium and carbon in cast steel (Figure 5.). Hence it may be judged that the austenitic cast steel stabilised with titanium is characterised by lower resistance to thermal shocks than the cast stabilised with niobium. At the same time, increasing carbon content in cast steel reduces in the presence of stabilisers its resistance to crack formation - Figure 5.b.

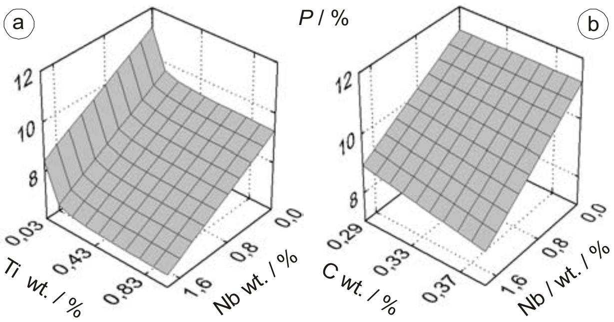


Figure 5. **Graphical illustration of the relationship shown by equation (3) for Ti = 0,03 %**
Slika 5. **Grafički prikaz odnosa prikazanog jednadžbom (3): za Ti = 0,03 %**

The cracks, observed in specimens, do not usually penetrate beyond the carburised layer (Figure 1.c). This proves that the non-carburised material impedes crack propagation - its ability to resist destruction is much stronger than that of a carburised layer. At the same time, the radial arrangement of cracks (Figure 2.), corresponding to a transcrystalline structure of the specimens suggests that grain boundaries are acting as regions of the preferred crack growth (Figure 6.).

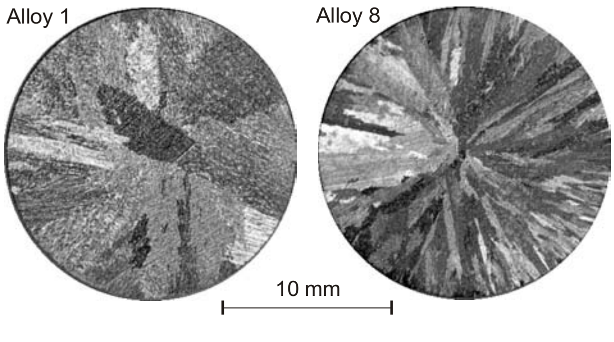


Figure 6. **Macrostructure of as-cast alloys**
Slika 6. **Mikrostruktura lijevanih legura**

The results of the measurements of mechanical properties (Table 4.) indicate that the effect of carburising atmosphere is particularly harmful in the case of the cast steel plastic behavior. At ambient temperature the austenitic cast steel after carburising behaves like a brittle material. An evaluation of the effect of niobium and/or titanium on the measured values of mechanical properties (equation (4) to (11), Figure 7.) mainly consists in the following statement: a separate or joint increase in the content of these elements usually reduces the value of $R_{0.2}$, $R_m$, KCV at both ambient temperature and at 900 °C, the adverse effect of titanium being usually much stronger than that of niobium. In the case of carbon and silicon one can observe their different

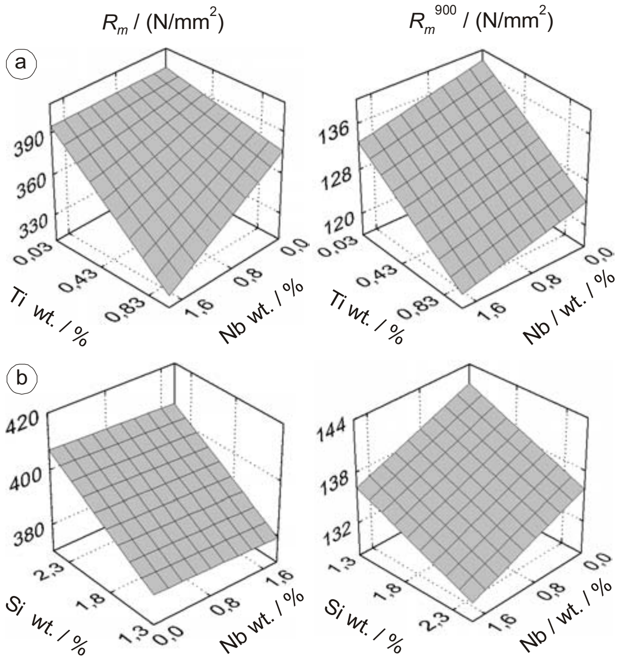


Figure 7. **Graphical illustration of the relationship shown by equations (5) and (8): a) for Si = 2,0 % and C = 0,35 %, b) for Ti = 0,03 %**
Slika 7. **Grafički prikaz odnosa prikazanih jednadžbama (5) i (8): a) za Si = 2,0 % i C = 0,35 %, b) za Ti = 0,03 %**

effects on the examined properties at ambient temperature and at 900 °C - equations (5), (8) and (9), Figure 7.b. This allows us to judge that an addition of silicon reduces the creep resistance of the examined cast steel.

$$R_{0.2} = 269,4 + 148,3\times C - 17,2\times Ti - 24,5\times Nb\times Ti$$
$$R^2 = 0,98,\ F = 68 \quad (4)$$

$$R_m = 353,4 + 21,7\times Si - 21\times Ti - 31,6\times Nb\times Ti$$
$$R^2 = 0,96,\ F = 32 \quad (5)$$

$$A_{10} = 0,72 + 3,08\times C + 0,25\times Ti$$
$$R^2 = 0,91,\ F = 24 \quad (6)$$

$$R_{0.2}^{900} = 109,1 - 78,6\times C - 3,2\times Ti$$
$$R^2 = 0,91,\ F = 26 \quad (7)$$

$$R_m^{900} = 172 - 67,2\times C - 4.9\times Si - 3,1\times Nb - 14,6\times Ti$$
$$R^2 = 0,98,\ F = 37 \quad (8)$$

$$A_{10}^{900} = 7,39\times Si + 5,18\times Ti$$
$$R^2 = 0,99,\ F = 277 \quad (9)$$

$$KCV = 26{,}7 - 12 \times Nb + 33{,}8 \times C \times Nb$$
$$R^2 = 0{,}93,\ F = 31 \qquad (10)$$

$$KCV^{900} = 30{,}5 - 11{,}4 \times Nb - 1{,}58 \times Ti + 31{,}2 \times C \times Nb$$
$$R^2 = 0{,}91,\ F = 14 \qquad (11)$$

## CONCLUSIONS

Expressed in general terms, the results of the conducted studies enable a statement to be made that additions of niobium and titanium reduce, with exception of the resistance to carburising, the examined utilisation properties of 0,3%C-30%Ni-18%Cr cast steel.

The collected experimental material and analysis of the results of measurements are basis for formulation of the following conclusions:

1. The resistance of the carburised cast steel to thermal fatigue measured by its tendency to the formation of surface cracks under the conditions of shock preheating and cooling decreases with increasing content of niobium and/or titanium as well as that of carbon. The results of measurements enable drawing of conclusion that austenitic cast steel stabilised with titanium is characterised by lower resistance to thermal fatigue than the cast steel stabilised with niobium.
2. Introduced separately or jointly, the stabilising elements usually reduce the mechanical and plastic properties of the carburised cast steel at ambient temperature and at 900 °C. The form of the determined quantitative relationships also points out to the following facts:
   - introduced jointly, the stabilising elements exert a much stronger adverse effect on the examined properties of cast steel than when introduced separately,
   - additions of niobium deteriorate the properties of cast steel to a smaller degree than the additions of titanium.

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