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# Mechanical properties of phase constituents in selected grades of cast steel

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## Abstract

The nanoindentation measurements performed on three cast steels of 0.3C-30Ni-18Cr type with various content of niobium and titanium were carried out. Mechanical properties of the main phase constituents of the alloys, such as austenite, MC and $M_{23}C_6$ type carbides, were determined and analysed. The values of hardness (H) and Young modulus (E) for the austenite matrix were similar within the tested alloys. Essential differences (H=12 ÷ 24 GPa; E = 250 ÷ 400 GPa) were found between the carbide phases present in tested alloys. The nanoindentation measurement on small particles is affected by different effects. One of these effects was excluded using numerical simulation of impressing the phase constituent into the matrix during indentation. The values of H and E obtained from simulation were: 30/450 GPa for NbC; 50/580 GPa for TiC; and 19/320 GPa for $Cr_{23}C_6$ respectively

**Keywords:** Nanoindentation, Hardness, Young modulus, Austenitic cast steel, Carbides

## 1. Introduction

Cast austenitic nickel-chromium steels are widely used in many high temperature applications. Their chemical composition has undergone various modifications aiming at improving the operating characteristics [1]. Introducing niobium or titanium to 0.3%C-30%Ni-18%Cr cast steel causes the formation in the as-cast structure of niobium or titanium MC carbides instead of chromium carbides of $M_{23}C_6$ type. Both niobium and titanium has beneficial effect on creep behaviour and also impede the process of corrosion in carburising atmosphere. However, they simultaneously contribute to the formation of intermetallic phases during exposure to high temperature and may adversely affect the plastic properties of cast steel, increasing its brittleness in service [1,2]. Each of the phases has its own hardness which makes their differentiation possible by the use of microhardness [3] or nanoindentation method [4].

In the recent years the nanoindentation method has become a valuable tool for the evaluation of materials mechanical properties. The method allows to determine the hardness and the elastic modulus from the nanoindentation load displacement data. The main advantage of this method is that the load applied can be very small and therefore the dimensions of measured microstructure elements (phase, particles) can be small and it is possible to determine their individual contributions in multiphase alloys. During the nanoindentation of phase constituents the measurement is affected by different effects. One of them, impressing of investigated constituent into the matrix during the indentation is confirmed using the numeric simulation.

In the present work the mechanical properties of phase constituents present in the microstructure of 0.3C-30Ni-18Cr cast steel with titanium and niobium additions were investigated by use of nanoindentation method.

## 2. Investigated material and tests

The austenitic 0.3C-30Ni-18Cr cast steel was used for investigations. The content of individual elements was varying within the chemical composition of the tested alloys and is shown in Table 1. The investigated alloys were in the same heat treatment condition - after annealing in air at 900°C for 500 hrs and then cooling down together with furnace.

Table 1.
The content of Ti and Nb in 0.3C-18Cr-30Ni cast steel [wt.%]

| Alloy no | Ti | Nb |
|---|---|---|
| 1 | 0.03 | 0,03 |
| 2 | 1.00 | 0.03 |
| 3 | 0.05 | 1.84 |

The phase composition was investigated by X-ray diffraction method. Detailed analysis of XRD diffraction patterns is presented in work [5]. The microstructure investigations were carried out with use of optical microscope, SEM and AFM microscopy

Nanoindentation experiments were carried out to determine the hardness and elastic modulus of matrix and phase constituents. Tests were performed using Nanoindenter XP with a diamond pyramidal-shaped Berkovich type indenter. The experiments were carried out by the CSM method at a constant depth of 300 nm. For each alloy a set of 100 indentations was performed. The measurements were taken on polished metallographic cross-sections of etched specimens.

### 2.1. Microstructure

Austenite matrix and phase precipitates compose the microstructure of tested alloys. Titanium and niobium (alloy 2 and 3 respectively) induce the refinement of microstructure of the base cast steel (alloy 1), Fig. 1. Both the elements Ti and Nb change the kind of precipitated eutectic carbides in tested alloys from $M_{23}C_6$ on MC type and therefore change the morphology of primary precipitates. The shape and distribution of carbides in the alloy varies depending on the kind of element, shown in Fig.1.

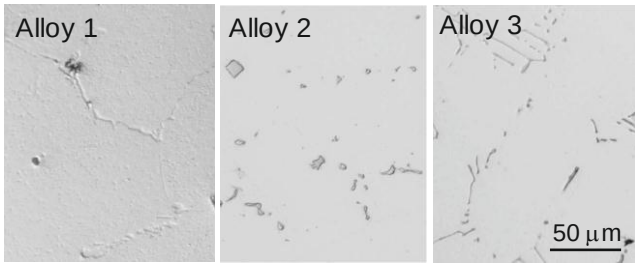

Fig. 1. Microstructure of tested alloys

The research carried out on deep etched samples of discussed cast steel revealed that the chromium carbides of $M_{23}C_6$ type are of rod shape, NbC crystallize as plates while carbonitrides TiCN form cubic shape [5].

Annealing of cast steel brings about the secondary precipitation processes. The small intermetallic phases precipitate in the matrix - see alloy 1 in Fig. 1.

Chromium carbides of $M_{23}C_6$ type are the only precipitates in alloy 1. They form almost continuous network at boundaries of dendrites. Carbides $M_{23}C_6$ are present in alloys 2 and 3 as well. Mostly they appear at the precipitate-matrix boundary, but the small precipitates of chromium carbides can also be seen in the matrix, as in Fig. 2.

The main phase constituents in alloy 2 are titanium carbides TiC and in alloy 3 niobium carbides NbC. Additionally carbonitride TiN which forms the center of TiC precipitates was found in very small quantities in alloy 2. In both alloys 2 and 3 the phase G of formula $Ni_{16}Ti_6Si_7$ and $Ni_{16}Nb_6Si_7$ respectively was also identified. Phase G rich in silicon and nickel was found surrounding some of the primary precipitates of MC carbides, as in Fig. 2

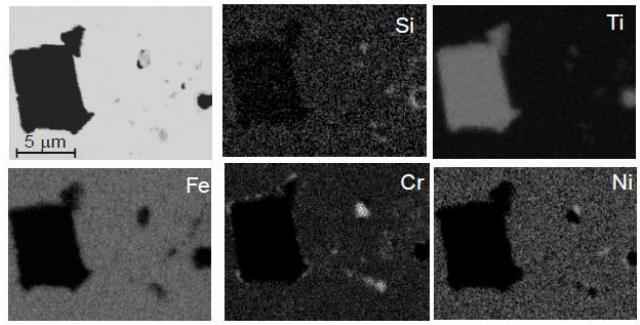

Fig. 2. Microstructure and mapping of elements in alloy 2, SEM

### 2.2. Nanoindentation and simulation

The indentations were done in the vicinity of interdendritic boundaries. The aim was to encompass the region of both the austenite matrix and eutectic carbides precipitates. Indentation of 100 points was perform with a step of 7,5 µm what gave 90 x 55 µm of measured area. Indentations were mostly located in the matrix. The piece of indentation grid in alloy 1 is shown in Fig. 3a. An indentations pointed in both matrix and carbides precipitates can be seen. The 3D -plot of the surface of indentation into the austenite matrix is shown in Fig. 3 b.

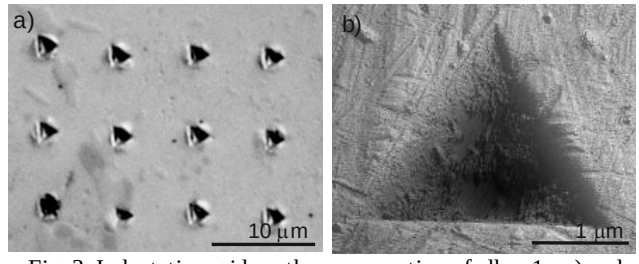

Fig. 3. Indentation grid on the cross section of alloy 1 - a) and AFM 3D surface plot of indentation into the matrix - b)

The results of nanoindentation experiments are presented in Table 2. The values of $E_{IT}$ and $H_{IT}$ for the matrix are in good agreement with literature data but the phase constituent values of $E_{IT}$ and $H_{IT}$ are relatively small and measured with high scatter.

From all measurements only 10% were pointed into the phase constituent (particle) and never were placed into the center of particle. The carbide precipitates are not precisely homogeneous due to their partial transformation into the G phase. Moreover the shape and size of

the particle underneath the measured surface is unknown and depends on basic shape of the particle, its orientation and cross section. Finally, an effective diameter of particles which resist penetration of indenter is unknown. All this could be the reason for the scatter, if the same phase is measured.

Table 2.
Mechanical properties of phase constituents of cast steel determined by nanoindentation

| | Matrix | | Phase particles | |
|---|---|---|---|---|
| **(GPa)** | $\mathbf{E_{IT}}$ | $\mathbf{H_{IT}}$ | $\mathbf{H_{IT}}$ | $\mathbf{E_{IT}}$ |
| **Alloy 1** | 209±10 | 4,2±0,45 | 13±6 | 264±37 |
| **Alloy 2** | 197±16 | 4,8±0,62 | 24±17 | 395±130 |
| **Alloy 3** | 203±10 | 4,3±0,56 | 12±5 | 283±16 |

Results presented in Table 2 are with high scatter. The question is what is the correct value and what affects the results? One of the many possible influences is impressing the particles into the matrix during the indentation. If the constituent is impressed into the matrix then the measured depth (h) is composed of:

$$h = h_i + h_h \tag{1}$$

where $h_i$ is an indentation depth of the constituent into the matrix and $h_h$ is an indentation depth of indenter into the constituent. Suppose that the effective radius of the constituent is big enough and the constituent is impressed into the matrix only elastically and indentation of the tip into the particle ($h_h$) is elasto-plastic, then :

$$h = h_{ie} + h_{he} + h_{hp} \tag{2}$$

After expressing [6] the eq. (2) we can achieve the following equation:

$$h = \frac{F}{2aE_{rm}} + \sqrt{F}\left(\left(\varepsilon\frac{\sqrt{H_c\pi}}{2E_{rc}}\right) + \left(3\sqrt{3}H_c\tan^2\theta\right)^{-\frac{1}{2}}\right) \tag{3}$$

In eq. (3) there are three unknown parameters: constituent hardness ($H_c$), constituent reduced modulus ($E_{rc}$) and effective radius ($a$) of the constituent. The effective radius is the minimum distance between the placement of indent and an edge of constituent. An iterative process was used to determine these parameters with following conditions: Iterations were done on all constituents measurements. The constituents have the same hardness and modulus, radius of the constituents can not exceed maximal length of the particle, the cylindrical shape of the constituents. The fitting was started at contact point and stopped at first inhomogeneity (pop-in, bending, etc.) on load-displacement curve. The correctness of eq. (3) without particle behaviour term was tested on three different bulk materials [7]. Calculated and measured data for all three materials are in good agreement.
The results of simulation using the eq. no (3) are presented in Fig. 4. Circle line symbolizes measured curve, dashed line is simulation of indentation into a bulk material with properties of the constituent, full line is simulation by the eq. (3). The fit meets the measured curve to first pop-in (step on the curve), which appears when crack or phase transformation starts. The difference between the dashed and measured curve describes influence of the constituent penetration into the matrix. It means that results are strongly influenced by impressing of the constituent into the matrix. The influence of the constituent size on hardness and modulus values is presented in Fig. 5. The properties measured on the biggest particles (the reciprocal value of radius goes to zero) could be supposed as the most realistic values of constituent of alloy 1. After the exponential fitting and extrapolating to infinite size of constituent we get values 19 GPa and 322 GPa for hardness and modulus values, respectively.

The same procedure was used for all three alloys (Fig. 6, 7) and the hardness and modulus results of main phase constituents are presented in Table 3

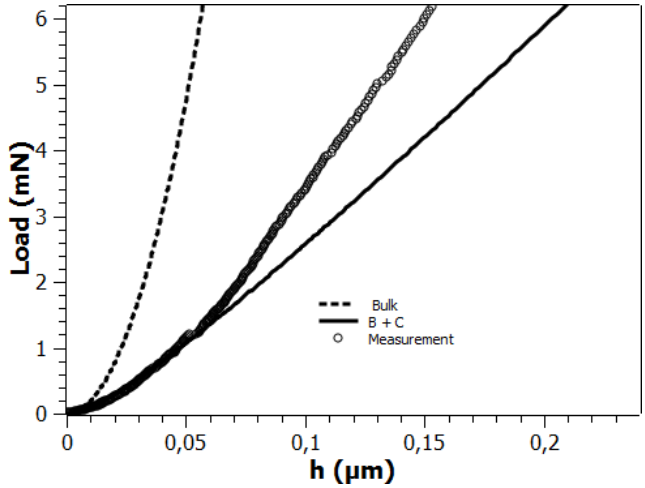

Fig. 4. Indentation curve of alloy 1 phase constituent (Measurement); simulation of bulk material with properties of particle (Bulk) and indentation of constituent with cylinder shape (B+C)

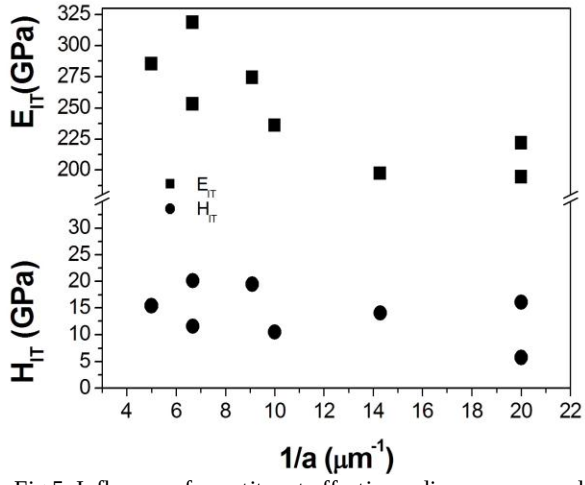

Fig 5. Influence of constituent effective radius on measured values of hardness and modulus, alloy 1

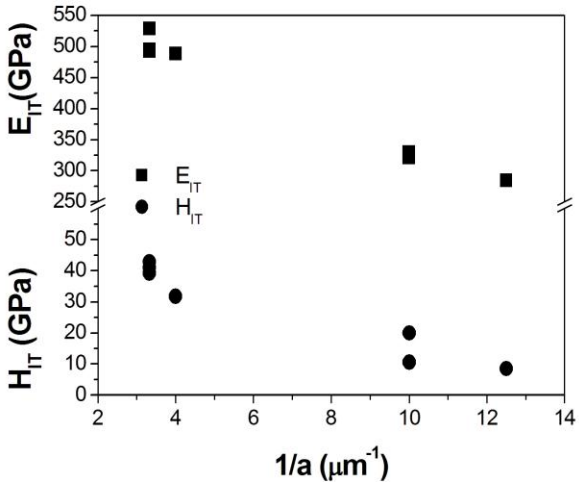

Fig 6. Influence of constituent effective radius on measured values of hardness and modulus, alloy 2

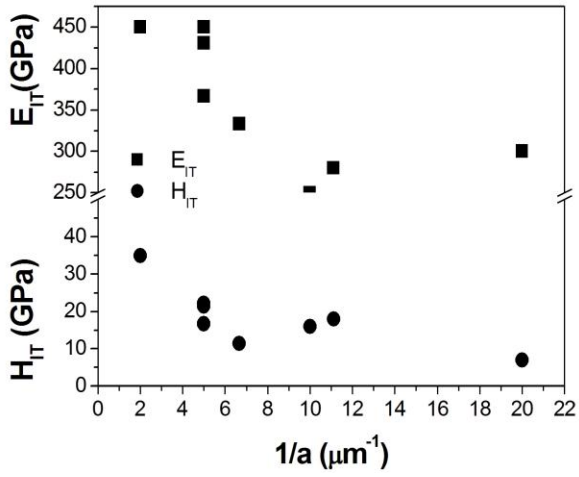

Fig 7. Influence of constituent effective radius on measured values of hardness and modulus, alloy 3

Table 3.
Properties of phase constituents determined by simulation

| (GPa) | Main phase constituents | $H_{IT}$ | $E_{IT}$ |
|---|---|---|---|
| **Alloy 1** | $M_{23}C_6$ | 19 | 320 |
| **Alloy 2** | TiC(N) | 50 | 580 |
| **Alloy 3** | NbC | 30 | 450 |

# 3. Conclusions

On the basis of nanoindentation measurements of alloys based on austenitic cast steel 0.3C-30Ni-18Cr with various content of niobium and titanium, the values of elastic modulus E and hardness H of phase constituents identified in alloys were determined and evaluated. The real values of $H_{IT}$ an $E_{IT}$ reach the maximum of measured values.

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