Effect of deformation parameters on microstructure evolution and properties of 33NiCrMoV15 steel

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Abstract. The paper deals with the effect of temperature and strain rate to microstructure evolution and final hardness of 33NiCrMoV15 steel after the simulation of the deformation process on real samples using of dilatometer DIL805D. The main goal of the simulation is the evaluation of Peak Stress – deformation resistance for different deformation temperatures and rates. Measured values are also processed by regression analysis to define the mathematical model of the monitored dependency. Examined steel is high strength middle alloyed steel with an optimal level of plastic properties used for high loaded parts including howitzers and tanks gun barrels.

1. Introduction
Investigated steel 33NiCrMoV belongs to the group of ultra-high strength low alloy martensitic steels. The steel combine high strength with good ductility, fatigue strength and creep resistance. One of the main applications of the steel is howitzer gun barrel production where the described properties are advantageously employed. The paper presents the evaluation of microstructure of the 33NiCrMoV steel after the application of deformation with selected specific rates from small to high and at temperatures from 800 °C to 1200 °C.

As is well known and described in the literature [1–3], application of the deformation to the primary austenitic grains before or during phase transformation of the austenite to the martensite or other phases has noticeable effects to the microstructure evolution and consequently to the final properties of a steel (figure 1).

Figure 1. Microstructure changes caused by deformation in three basic temperature areas [4].
Application of the deformation brings effect at both high and lower temperatures. If the deformation is applied at high temperatures with recrystallization effect, grain refinement occurs due to the repeated recrystallization. More noticeable effect to microstructure occurs at lower temperatures in the uncrystallization area (figure 1). The grains remain deformed and the deformation strips are formed inside them. Consequently, new phase (martensite) nucleates not only as a standard at grain boundaries \( \gamma \), but also inside the grain at the points of deformation strips. Nucleation within the deformed austenitic grains is one of the most important aspects of thermo-mechanical processing \([4, 5]\).

2. Experimental procedure

2.1. Experimental materials
The base material used for this paper is a middle alloyed 33NiCrMoV15 steel used in weapon industry. Raw material was cast as bars subsequently forged, vacuum remelted and heat treated by quenching and tempering. At first, chemical composition of selected steel was verified. CCD-based Spectro Jr CCD optical emission spectrometer was used to measure chemical composition of the steel (table 1). Mechanical properties of experimental materials were also verified. Vickers hardness measurement (HV5) of experimental samples was performed, using Instron-Wolpert testing device with loading 49.03 N and indentation time \( t = 10 \) s (EN ISO 6507-1). Standard tensile strength test (EN ISO 6892-1) was performed to acquire strength characteristics, while the standard Charpy Impact test (EN ISO 179-1) was used to measure toughness. All mechanical properties acquired by these measurements are summarized in table 2. Experimental samples in the shape of a cylinder with dimensions Ø5x10 mm were made of the experimental steel which were subsequently subjected to dilatometric analysis (chapter 2.2.).

| Table 1. Chemical composition of experiment steel (wt. %). |
|---|---|---|---|---|---|---|---|
| C | Mn | Si | Cr | Ni | Mo | V | Fe |
| 0.409 | 0.558 | 0.381 | 0.926 | 2.855 | 0.221 | 0.1363 | Bal |

| Table 2. Basic mechanical properties of experimental steel. |
|---|---|---|---|---|
| Tensile strength \( R_m \) (MPa) | Proof stress 0.05 \( R_{p0.05} \) (MPa) | Toughness \( KCU \) at 20 °C | Hardness (HV) | Elongation \( A_5 \) (%) |
| 1500 | 1079 | 23 | 500 | 20 |

2.2. Treatment of experimental samples and deformation analysis
Dilatometer DIL805D was used to simulate the treatment of the samples in real conditions. The sample is inserted to the work chamber of the dilatometer and then subjected to the programmed thermal and deformation cycle. Heating phase of the cycle is performed by induction heating under vacuum and cooling phase with using of helium as protective and cooling gas. The deformation of the sample is provided by its compression with using the piston installed in the chamber.

The result from the deformation analysis is the deformation curve – dependency between true strain \( \varphi (-) \) and true stress \( \sigma \) (MPa) belonging to the specific test parameters. Main characteristic evaluated form the curve is the peak stress \( \sigma_p \) (MPa) – maximal value of the stress in the curve which is also considered as the deformation resistance of the material \([6, 7]\).

There are used equal heating parameters for all experimental samples consist of slow heating (1 °C s\(^{-1}\)) to the austenitizing temperature \( T \) where following temperatures: 900 °C, 1000 °C, 1100 °C and 1200 °C with holding period (1800 s) to provide complete austenitizing of sample volume were used. The processing then continued with deformation phase where eight different deformation rates \( \varphi' \) were used.
from very rapid to slow (0.001 s\(^{-1}\), 0.01 s\(^{-1}\), 0.1 s\(^{-1}\), 1 s\(^{-1}\)). Used treatment is depicted in figure 2. As a result of this processing, sixteen different combinations of the experimental samples were prepared.

![Figure 2. Treating scheme of experimental samples.](image)

2.3. Microstructure analysis and hardness measurements
The microstructure of each experimental samples previously subjected to the specific treatment in dilatometer was subsequently observed by optical microscopy. The samples were treated by a standard metallographic procedure consisting of grinding, polishing and etching by Nital (3% HNO\(_3\) in ethanol) to emphasize the microstructure. After that, the hardness of all samples was evaluated by standard Vickers hardness test with \( F = 49.03 \) N load force and \( t = 10 \) s indentation time. The samples were indented in the center line to measure the core hardness and final hardness of the sample was evaluated as the average value from three measurements.

3. Result and discussions

3.1. True strain - true stress curves
There were measured all true stress - true strain curves for every combination of temperature and deformation rate with an evaluation of maximal value – peak stress \( \sigma_p \). The example of the curves for \( \phi' = 1 \) s\(^{-1}\) is in figure 3.

![Figure 3. True stress - true strain curve for \( \phi' = 0.1 \) s\(^{-1}\).](image)
3.2. Mathematical modeling of dependency of peak stress to temperature and deformation rate

As the all measured curves show, the peak stress is related both to temperature and deformation rate. This dependency could be expressed by Garofallo equation [8, 9], describing the dependence of deformation rate to other parameters:

\[ \varphi' = C \left[ \sinh (a \sigma_p) \right]^n \exp \left( -\frac{Q}{RT} \right) \]  

where \( \varphi' (s^{-1}) \) is deformation rate, \( T (K) \) – deformation temperature, \( \sigma_p \) (MPa) – peak stress, \( Q \) (J mol\(^{-1}\)) – activation energy, \( R \) (J K\(^{-1}\) mol\(^{-1}\)) – universal gas constant (8.314 J K\(^{-1}\) mol\(^{-1}\)) and \( n \) (-), \( C \) (s\(^{-1}\)), \( a \) (MPa\(^{-1}\)) are material constants specific for deformed material.

The peak stress \( \sigma_p \) can be expressed from equation (1):

\[ \sigma_p = \frac{1}{a} \text{arcsinh} \left( \left[ \frac{\varphi'}{C} \exp \left( -\frac{Q}{RT} \right) \right]^\frac{1}{n} \right) \]  

Two parametric regression model could be defined with using of equation (2) based on experimental data. The input independent variables are deformation temperature \( T \) with deformation rate \( \varphi' \) and the dependent variable is peak stress \( \sigma_p \). R is universal physical constant and other equation elements are considered as regression constant to be calculated. In result, it is necessary to solve two parametric regression problem:

\[ \sigma_p = f(T, \varphi') \]  

There are exist several methods how to solve a regression problem. In this case, Scilab regression tools was used to solve the problem [10]. The Script with about hundred lines long was programmed to provide needed calculation and visualisation of the result data. The calculation of regression coefficients is based on least squares methods. The comparison of measured and calculated data is in figure 4.

![Figure 4. Dependency of peak stress \( \sigma_p \) to deformation temperature \( T \) and rate \( \varphi' \).](image)

Coefficient of determination of the regression is \( R^2 = 98.6 \). Resulting regression coefficients values are shown in table 3.
3.3. Evaluation of microstructure

There is shown evaluation of microstructure in relation to deformation temperature and rate in figure 5. Only cases with limit values of \( T \) and \( \phi \) are presented in the picture. Slight change in grain size occurs after deformation at high temperatures where the recrystallization effect is not present (figure 5c versus 5d). Figure 5 corresponds to the case of minimal peak stress \( \sigma_p \) (deformation resistance).

![Figure 5](image)

**Figure 5.** Evaluation of microstructure in dependency to deformation temperature \( T \) and rate \( \phi \).

Most noticeable effect to microstructure is seen in case of high deformation rate (1 s\(^{-1}\)) and low temperature (900 °C). It is temperature range without recrystallization effect. The martensitic microstructure is very grain refined due to deformation of austenitic grain prior martensitic transformation and formation of deformation strips inside grains.

4. Conclusions

Several deformation analyses were performed using DIL805D in order to measure true stress - true strain curves (\( \sigma - \phi \)) for different deformation conditions – temperature and rate. The main evaluation criterion is the peak stress \( \sigma_p \) (deformation resistance). Measured values of the peak stress \( \sigma_p \) were then analysed.
in order to map its dependency to deformation temperature $T$ and deformation rate $\phi'$ and define the appropriate mathematic model. Regression analysis with using Scilab language was used to solve the problem. The analysis shows two important conclusions. Deformation resistance increases linearly in relation to decreasing deformation temperature. Also, deformation resistance increases exponentially with the strain rate.

In terms of microstructure, the high value of the deformation rate (1 s$^{-1}$) at low temperature (900 °C) has the greatest effect on its change. Under these conditions, a very fine-grained microstructure is formed with high values of mechanical characteristics as is hardness and strength.

5. References

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