STRAIN AND STRAIN AGING CHARACTERISTICS OF LOW-CARBON MICROALLOYED HEAVY PLATE STEEL GRADE S460MLO WITH PRE-QUALIFICATION FOR ARCTIC AREAS

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Abstract

In this work, the influence of strain and strain aging parameters on the strength and low temperature toughness of low-carbon microalloyed Nb-V-Ti and Nb-Ti steels is investigated. Industrial samples of 23 mm heavy plates after TMCP (Thermo-Mechanical Controlled Processing) and additional sPWHT (subsequent Post-Weld Heat Treatment) were used for the research. Theoretical foundations of steel aging were considered, and its primary parameters antimed mechanisms were described. One of the main challenges with steels used in Arctic areas is to ensure adequate material toughness to prevent brittle fracture. Therefore, a parameter indicating the susceptibility of steels to aging was determined. The change in the basic strength properties under 5 %, 7.5 % and 10 % pre-strain was determined. Subsequent aging was carried out by heating to 250 °C for 60 minutes. Impact tests were conducted at temperatures as low as -80 °C to determine the reliability and durability of constructions, especially in Arctic conditions. The presented results have been used with a positive outcome in the certification tests for production of heavy plate products in quality categories S355MLO, S420MLO, S460MLO according to the standards EN10225-1:2019 and NORSOK M-120, including Arctic editions (Annex F).

Keywords: low-carbon steel, TMCP, heavy plates, aging, mechanical properties

1. INTRODUCTION

The study focuses on investigating the impact of strain and strain ageing conditions on the mechanical properties of resilient low-carbon steel S460MLO in TMCP and TMCP+PWHT delivery condition. This is crucial for ensuring the long-term reliability of structures in offshore environments, particularly in subarctic and arctic conditions. Understanding ageing mechanisms in steel, characterized by changes in mechanical properties over time without visible microstructural alterations, is essential [1]. These processes, including deformation ageing, thermal ageing, and thermo-deformation ageing, are primarily influenced by the presence of carbon and nitrogen atoms and their interaction with newly formed dislocations [2]. The study aims to quantify the effect of applied strain on strain ageing, considering the diverse structural characteristics and alloying features of low-carbon steels. By elucidating the relationship between these factors and mechanical properties, the research contributes to enhancing the durability and reliability of offshore structures.

2. MATERIALS AND METHODS

The steels of two alloying systems of BOF production cast into 355 mm thick continuous cast slabs were used as the material of investigations. The chemical composition of the studied steels is in **Table 1**.

Table 1 Chemical composition of investigated steels, wt%

Steel	Alloying system	C	Mn	Si	S	P	N	Ni	Cev
A	Nb-V-Ti	0.06	1.45	0.24	0.001	0.005	0.004	0.2	0.33
B	Nb-Ti	0.04	1.44	0.18	0.001	0.008	0.005	0.2	0.31

Slabs were rolled on the reversing mill DanSteel 4200 [3] by two-stage thermomechanical rolling to a final plate thickness of 23 mm with the end of deformation in the γ+α-region. The level of basic mechanical properties for both steel grades corresponds to rolled products of quality category S460MLO.

A series of samples were taken from each of the rolled plates of appropriate chemical composition for mechanical testing. Half of the samples were heat treated by heating at 55 °C/hour to 580±3 °C, holding for 60 minutes and cooling to 400 °C at 55 °C/hour. This procedure simulates the heat treatment of assemblies or parts of the structure after assembly or repair welding (sPWHT - Simulation of post weld heat treatment). To simulate strain processes, specimens from both groups were subjected to tensile strain of 5 %, 7.5 % or 10 % (±0.5 %). The deformation was carried out at room temperature. Thermo-aging was simulated by heating of some samples to 250 °C and holding time for 60 min and then cooling on the air.

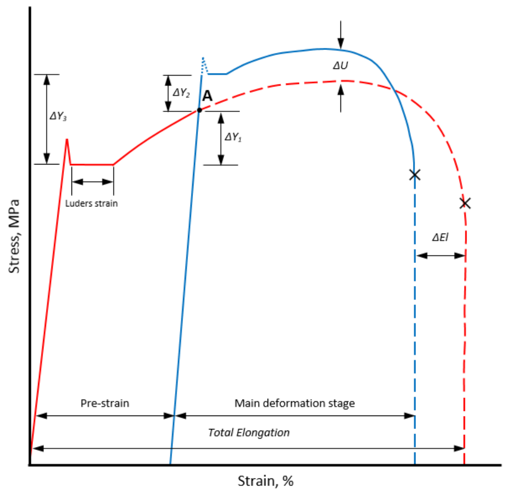


Figure 1 Diagram of changes in the main strength parameters in the stress-strain system at the stages of initial and main deformation

To visualize [4] the influence of ageing processes on the strength properties of steel, we used the stress-strain system (**Figure 1**) with a stress-strain diagram of initial loading up to point A corresponding to 5 %; 7.5 % or 10 % (±0.5 %) of pre-strain, and a tensile diagram after the main loading cycle for all the states under study. The following parameters were determined from the stress-strain diagram: ΔY_1 - change in yield strength $Rp_{0.2}$ due to pre-strain; ΔY_2 - change in yield strength Rp0.2 due to strain/stain age; ΔY_3 - change in total yield strength Rp0.2 due to pre-strain and strain/stain age; ΔU - change in tensile strength R_m due to pre-strain and strain/stain age; ΔEl - change in total elongation due to pre-strain and strain/stain age.

After preloading and subsequent unloading, some part of the stress-strain curve up to point A, corresponding to plastic deformation, remains in the diagram. If the sample is immediately retested, the curve shows an extended elastic region up to point A, and then extends exactly as if no stress drop had occurred during unloading. Under these conditions, no increase in yield strength is observed during the second loading cycle.

If the specimen is unloaded from the testing machine, i.e. aged either at ambient or elevated temperatures, the conditional yield stress $Rp_{0.2}$ is observed again during the second loading cycle, and its values are at a higher level than the flow stress prevailing at the end of the pre-deformation operation up to point A. This increase in the level of the yield stress $Rp_{0.2}$ after ageing is the most striking sign indicating ageing processes [5].

Tensile tests were carried out on transverse flat samples according to ISO 6892. For microstructural studies Carl Zeiss Axio Observer 7 MAT optical microscope was used. The standard method of etching with 5 % Nital acid was used to identify the microstructure.

3. DISCUSSION

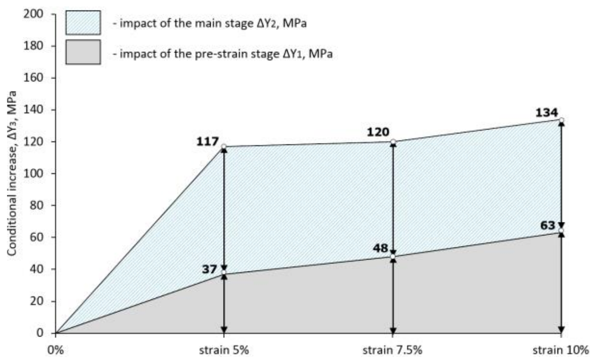
The steels under investigation are characterised by homogeneous ferrite-perlite microstructure without banding. In Steel A ~ 91 % of ferrite and 9 % of pearlite, and in Steel B the volume fraction of ferrite is higher - about 95 % and pearlite 5 %. The ferrite grains are fine, deformed and elongated in the rolling direction. The distribution of the diameter of the average conditional ferrite grain is quite stable and for Steel A in 1/8 thickness ~ 7.5 μm, in 1/4 ~ 8.3 μm, and in 1/2 - not more than 10 μm, for Steel B has 8.1 μm, 9.6 μm and 11.7 μm in 1/8, 1/4 and 1/2 thickness respectively.

The decrease in yield strength $Rp_{0.2}$ and tensile strength R_m is characteristic of the steels after undergoing additional heat treatment, reflecting the reduction in strength due to the partial alleviation of internal stresses during the applied heat treatment processes. After testing in the initial state for the investigated steels an upper yield point was observed in the tensile diagram the value of which is on average about 25-30 MPa.

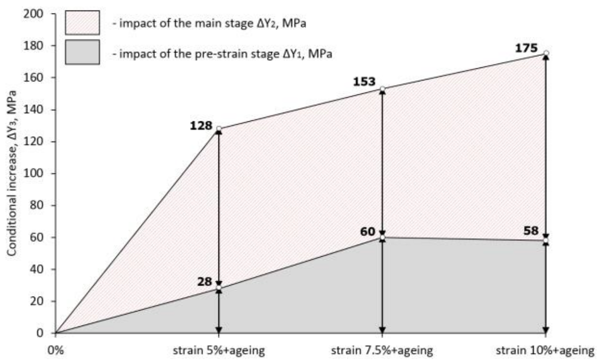
4. STRAIN AND STRAIN AGEING

To assess the susceptibility of the studied steels to aging, a pair of diagrams representing the preliminary-main stage of deformation was interpreted for each presented state based on the parameters in **Figure 1**. From these interpretations, the values of the main strength characteristics were determined, including the yield strength $Rp_{0.2}$ and its increase (ΔY_1, ΔY_2, ΔY_3), tensile strength Rm and its changes relative to the initial state ΔU, as well as elongation El and its deviations ΔEl.

According to aging sensitivity parameter ΔY_3 decomposed into two components (ΔY_1 from pre-deformation and ΔY_2 from aging followed by main deformation), diagrams were created. These allow evaluation of changes in yield strength between deformation stages, considering aging parameters and mechanism, and visualize the contribution of each component. In **Figure 2** for Nb-V-Ti steel, plots show conditional yield strength increase versus aging parameters. It is demonstrated that ΔY_3 consistently rises with greater pre-strain deformation: 117 MPa - 120 MPa - 134 MPa for Steel A, and 72 MPa - 110 MPa - 139 MPa for Steel B. Additional heating and holding elevates the increment: 128 MPa - 153 MPa - 175 MPa for Steel A, and 133 MPa - 147 MPa - 176 MPa for Steel B.



a



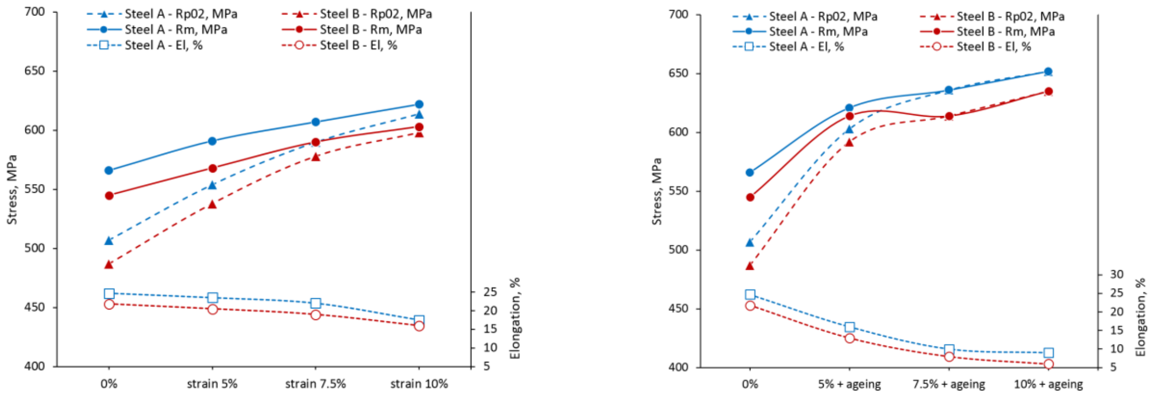
b

Figure 2 Conditional increase in yield strength in Steel A depending on ageing conditions: a - strain, b - strain ageing

The influence of pre-deformation on total yield strength increment remains consistent for both alloying systems. This component's contribution increases with higher pre-deformation percentages. For Steel A, at 5 % strain, it ranges from 22-32 %, at 7.5 % it is 39-40 %, and at 10 %, it reaches 33-47 % of the total increase. Steel B shows similar trends: 21-36 % at 5 % strain, 37-45 % at 7.5 %, and 31-41 % at 10 centum%. The main part of the increase is attributed to the second component ΔY_2, reflecting the influence of aging processes. Regardless of alloying and aging mechanism, ΔY_2 contribution is 2-3 times higher than ΔY_1.

The reappearance of the yield point in stress-strain diagrams occurs only after pre-deformed samples undergo heating before main deformation, indicating strain aging. This is due to new dispersed particles, acting as centres for blocking dislocations, and partial recovery processes, where fixed atoms move from dislocations to embedding positions. Aged material may contain increased dislocations, contributing to ΔY_3 and final strength properties [5, 6].

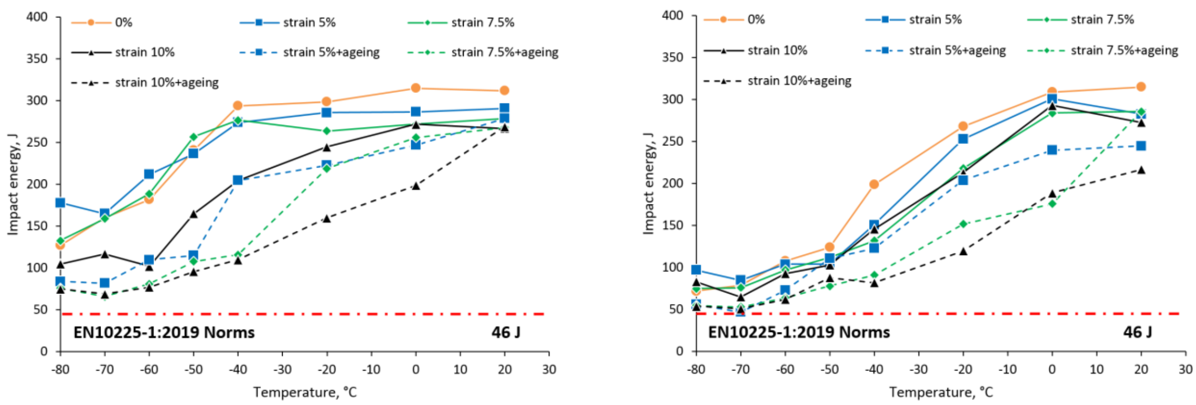
Comparing mechanical properties after aging with initial conditions helps assess changes in real structures over time. **Figure 3** illustrates variations in strength characteristics based on preloading and aging mechanism.



A b

Figure 3 Strength characteristics of the investigated steels after TMCP: a - strain, b - strain ageing

In steel A of the Nb-V-Ti alloying system, with increasing pre-deformation, an increase in the conditional yield strength $Rp_{0.2}$ and tensile strength R_m is observed: 514 MPa and 566 MPa at initial condition, 554 MPa and 591 MPa for 5 % strain, 590 MPa and 607 MPa for 7.5 % strain, 614 MPa and 622 MPa for pre-applied 10 % strain. Similarly, for Steel B, which belongs to the Nb-Ti alloying system, the trend in strength properties continues, showing a similar pattern of increase from the initial state to 5-7.5-10 % pre-strain. In this case, $Rp_{0.2}$ is distributed as: 509-538-538-578-598 MPa, and R_m: 545-568-590-603 MPa.



A b

Figure 4 Dependence of impact energy of steels after TMCP on the test temperature at the investigated ageing parameters: a - Steel A, b - Steel B

In strain aging (**Figure 3a**), the trend of increasing values persists. In Steel A, $Rp_{0.2}$ increases from 514 MPa in TMCP delivery condition to 603-636-652 MPa, and in Steel B from 509 MPa to 592-614-635 MPa. R_m changes from 566 MPa to 621-636-652 MPa in Steel A and from 545 MPa to 614-614-635 MPa in Steel B.

Relative elongation decreases compared to the initial state, reaching 6-7 % in strain aging and up to 16 % in strain aging with subsequent heating.

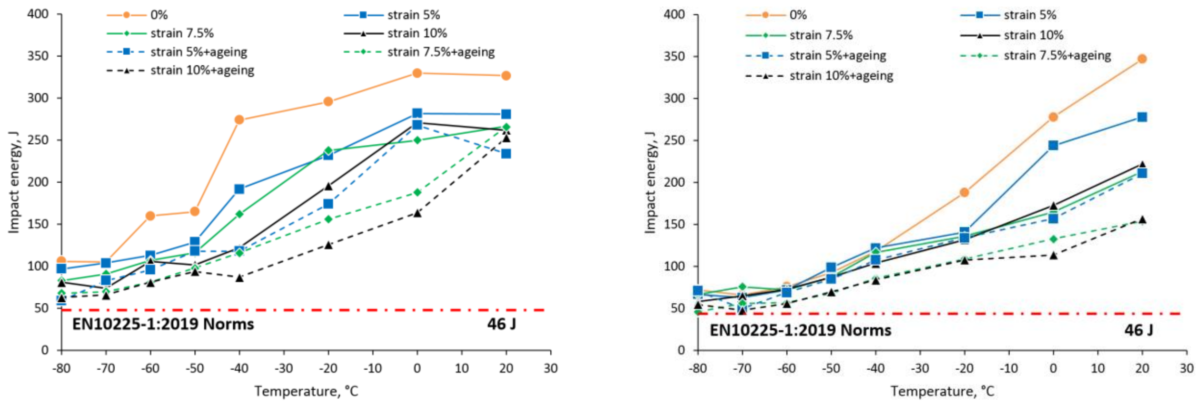
Figure 4 shows sequential curves of impact work changes at test temperatures from 20 °C to -80 °C after strain and strain ageing. Studies have indicated that aging processes in mild steel result to an increase in the ductile-brittle transition temperature with higher deformation percentages. In this study, a decrease in impact toughness was observed after pre-deformation, and subsequent ageing at 250 °C for 60 minutes further reduces impact energy for both types of steels. The overall impact energy of Nb-V-Ti steel is slightly higher than that of Nb-Ti steel, particularly noticeable at test temperatures of -50 °C and below.

Analysing the results, it is important to note the low nitrogen content in the steel, mostly bound by nitride-forming elements. Thus, carbon located in pearlite as carbide, plays a significant role in terms of chemical composition. Most of the carbon capable of interacting with dislocations is expected to be in ferrite [7].

5. EFFECT OF POST WELD HEAT TREATMENT

For various industries (oil, gas, chemical) it is mandatory to evaluate the resistance of steel to ageing processes after additional heat treatment, which manufacturers of steel structures do after welding operations. The purpose of this treatment is to alleviate residual stresses generated during welding, which could adversely affect the operational properties of the welded joint [4]. Values of the main strength parameters and their variation with respect to the initial condition were obtained from the diagrams of the preliminary and main deformation stages, interpreted with reference to the **Figure 1**. The influence of the pre-deformation stage ΔY_1 on the total yield strength increase ΔY_3, like the TMCP condition, depends on the percentage of strain applied at this stage. It consistently increases, ranging from 35 MPa with 5 % strain to 72-74 MPa with 10 % loading, irrespective of the steel type.

The significance of the main stage, i.e., the percentage of the ΔY_2 component of the total increase in $Rp_{0.2}$, in the case of strain, is on average 56 % for Steel A and 52 % for steel B, while in the case of strain ageing, it is 57 % and 64 %, respectively. The contribution of ΔY_2 is slightly lower than after TMCP, which may be due to partial stress relief in the process of de-stressing caused by the additional PWHT.



A b

Figure 5 Dependence of impact energy of steels after TMCP+PWHT condition on the test temperature at the investigated ageing parameters: a - Steel A, b - Steel B

The total conditional increase ΔY_3 during strain varies with deformation: for Steel A, it ranges from 89 MPa (5 %) to 157 MPa (10 %), and for Steel B, it ranges from 82 MPa to 149 MPa, respectively. Upon aging with subsequent heating, the reappearance of the yield point is observed. For Steel A, the ΔY_1 increment ranges from 121 MPa to 180 MPa, and for steel B, it ranges from 139 MPa to 165 MPa. For Steel A, $Rp_{0.2}$ increases from 480 MPa to 641 MPa, R_m from 556 MPa to 641 MPa, and El decreases from 26.5 % to 7 %. For Steel B, Rp0.2 increases from 466 MPa to 612 MPa, Rm from 535 MPa to 632 MPa, and El decreases from 25 % to 7.5 %. The observed rapid decrease of $Rp_{0.2}$ and R_m values in the state without aging can be related to the de-hardening that occurred during additional heat treatment at 580 °C and is caused by the decrease of dislocation density, release of dispersed particles, return processes, as well as almost complete stress relief [8]. **Figure 8** illustrates impact energy curves for TMCP+PWHT steel across temperatures from 20 °C to -80 °C. Steel A maintains high impact energy levels after TMCP (**Figure 4a**) and TMCP+PWHT (**Figure 5a**) up to -40 °C, declining with aging severity. Below -40 °C, there is a gradual decrease, occasionally with spikes in the ductile-brittle transition. Steel B exhibits lower impact energy, with stable distribution up to -40 °C only in TMCP (**Figure 4b**). For TMCP+PWHT (**Figure 5b**), curves sharply decline from 20 °C, narrowing the spread across tested states from -40°C to -80 °C. The EN10225-1:2019 standard regulates the norms for only 5 % pre-deformation. The steels studied fulfil these criteria in all tested states.

6. CONCLUSION

The variations between the main strength characteristic and the aging sensitivity parameter ΔY_3 have been determined for low-carbon microalloyed steels according to EN10225-1:2019 quality grade S460MLO of 23 mm thickness in TMCP and TMCP+PWHT delivery conditions. Results showed that for the investigated Nb-V-Ti and Nb-Ti steels, preliminary 10 % deformation increases $Rp_{0.2}$ by ~15-20 % at ΔY_3 130-140 MPa.

At -40 °C impact energy is by ~25-35 % reduced. Heating up to 250 °C and holding for 60 minutes additionally increases $Rp_{0.2}$ of the investigated steels by ~10-15 % at ΔY_3 170-180 MPa.

The impact energy values are consistently reducing by ~30-35 %. After subsequent PWHT, the tendency for an increase in $Rp_{0.2}$ remains, but impact energy decreases by ~60-70% from the initial level.

Summarizing the test results, investigated Nb-V-Ti steel has a higher resistance to aging processes and can be recommended for production of steel grade S460MLO used for critical marine applications. The tests revealed the reappearance of the upper yield point after strain ageing and can be explained by the appearance of new dispersed particles, which became centres for blocking dislocations because of partial return processes, when a part of already fixed atoms moved from dislocations to embedding positions.

The results of the research were applied in passing with positive results the certification tests of thick plates of quality category S460MLO, S420MLO, S355MLO according to EN10225-1:2019 and NORSOK M-120 standards, including the Arctic version.

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