HIGH STRENGTH STEEL BEHAVIOUR AT TENSILE TEST

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Abstract

The paper focus on possibilities of imaging microstructure changes in high strength low-alloyed carbon steel using an in-situ tensile stage placed in scanning electron microscope (SEM). The sample can be observed either in secondary electrons (SE) or by electron backscattered diffraction (EBSD). We used both types of imaging for different purposes: SE were used mainly for observation of crack initation and propagation and EBSD was used for grain deformations observation. Both methods need different specimen preparation and moreover we used different specimen shapes. All experiments were performed with several TRIP (transformation-induced plasticity) steels containing 0.2 - 0.4 % C, 0.5 - 2 % Si, 0.6 - 1.5 % Mn, 0.03 - 0.06 % Nb.

Keywords: Steel, microstructure, electron microscopy, tensile testing, in-situ testing

1. INTRODUCTION

Possibilities of imaging microstructure changes using scanning electron microscope (SEM) equiped with an in-situ tensile stage are studied in the paper. Tensile testing provides number of information about physical behaviour of material [1-4]. But these parameters are obtained from the correlation of state of the sample before and after the experiment. To see which processes take place in the matter of the sample during the one need to interrupt the experiment, remove the sample from tensile testing machine and prepare a metallographic specimen which cannot be used for tensile testing again. For observation of continuous changes which occurs during tensile testing one must make a series of experiments with the same parameters and to end each test continuously e.g. at different load. If the tensile test machine is already placed in SEM, it is possible to interrupt the experiment, analyse the microstructure and continue with the same sample, this can save both material and time [5].

In spite of its great potential for delivering extremely useful experimental information [6,7], the combination of the two techniques (in-situ tensile testing and EBSD) is still not extensively used due to technical challenges posed by drift, hardware design limitations and acquisition time [8].

2. MATERIALS AND METHODS

High strength low-alloyed carbon steels were used for experiments. Samples were made from various low-alloyed steels with 0.2 - 0.4 % C, 0.5 -2 % Si, 0.6 - 1.5 % Mn, 0.03 - 0.06 % Nb which were treated with several schedules of thermo-mechanical treatment. All examined steel possessed good properties as high ductility, toughness and suitable microstructure. Resulting microstructures consisted of the mixture of free ferrite, bainite and retained austenite [5]. Samples were water jet cut from the steel rods and then grinded, polished in a standard way of a metallographic sample up to 1μm diamond paste. Prior to testing by SE, samples were etched in 3 % Nital solution. For EBSD analysis sample preparation consisted again from mechanical grinding and polishing but moreover it had to be electrolytically polished with a Struers electrolyte A2 based on perchloric acid. Electrolytic polishing took place on Electropol-5 from Struers, parameters of the polishing were as follows: 19 V, 15 s, 16 flow rate, 0.15 A. Scanning electron microscope Zeiss EVO MA25 equipped with LaB_6 cathode, EDS (SDD X-Max^N 20) and EBSD (NordlysNano) detectors both from manufacturer Oxford

Instruments, was used. Parameters for SE imaging were typically 15 kV EHT, WD 21 mm, for EBSD EHT was 20 kV, WD 55 mm, sample was tilted 70° against the EBSD detector. Very long working distance is necessary to allow manipulation with the tensile stage in the SEM.

Tensile stage MTII/SEMTester 1000EBSD which is compatible with mentioned SEM was provided from MTI Instruments. It can develop loading force up to 4500 N using variation of loading velocity (0.02 to 2 mm / s). The tensile stage, or more precisely a sample loaded in the tensile stage can be heated up to 1200°C, however all the experiments in the study took place under normal temperature. Control of the tensile stage is operated via software MTESTQuattro ™ from Admet Inc. Software which allows required set-up of the tensile experiment and also enables measurement of selected parameters. Samples used for the tensile stage are of two different geometries (**Figure 1**) both have the same outer dimensions (45 x 10 mm) but differ in the gauge section. These differences are made for the purpose of observation. Classical dog-bone shaped specimen is used for test focused on mechanical properties and grain deformation tests (EBSD). The rounded one is used for SE tests as the shape of the specimen ensures crack propagation right in the middle of the gauge section while EBSD samples brakes closer to one of the shoulder.

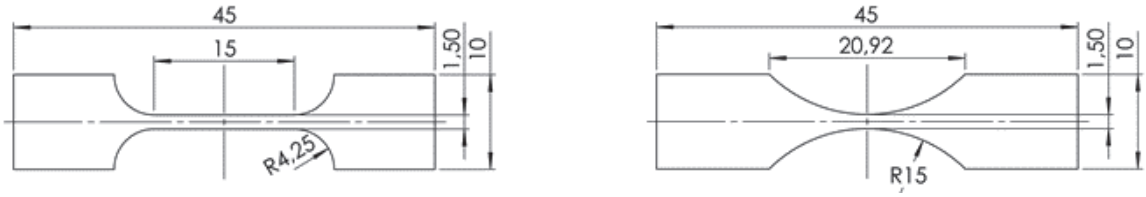


Figure 1 Sample shapes and dimensions used for in-situ tensile tests: plain (left) and curved (right)

These dimensions, alongside with sample surface preparation (grinding, polishing, etching) resulted from previous work for the test being long enough for SEM observation and it is ensured that each sample reaches its limit and is fully broken at the end of the test.

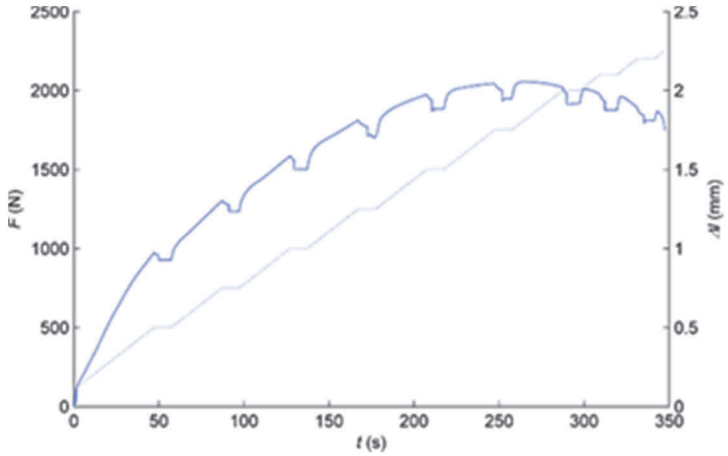


Figure 2 A time evolution of load *F* and elongation Δl in tensile test with pauses for EBSD

2.1. Principle of the testing

At first, one sample of examined material was tested outside the microscope on air without interrupting to calculate stress-strain curves and parameters like engineering stress and strain. Then a new, freshly polished and etched sample was mounted into the jaws of tensile stage which was then placed in a SEM and connected with a controller. The test itself was controlled by position of the jaws (velocity 0.5 mm / min) with several pauses for microstructure analysis, the jaws shift was interrupted, which was followed by 15 s relaxation of

material. Within this period, to prevent data log overflow, the sampling was paused manually allowing enough time (10 - 20 minutes) for EBSD maps or SEM images to be acquired. These manual pauses caused the sharp drops in the measured load in the scheduled pause period. (**Figure 2**) Imaging interruptions cover all region of stress-strain curve - see **Figures 3, 4**. Monitored parameters were: load and absolute elongation. After the break of the sample, it was dismounted from the tensile stage and the fracture was examined. Images or maps from particular elongation pauses were compared to illustrate the deformation.

3. RESULTS AND DISCUSSION

Experiments focused on crack initiation and propagation are already well mastered starting with sample preparation to the sample break and can provide nice view of the microstructure changes and deformation of the material.

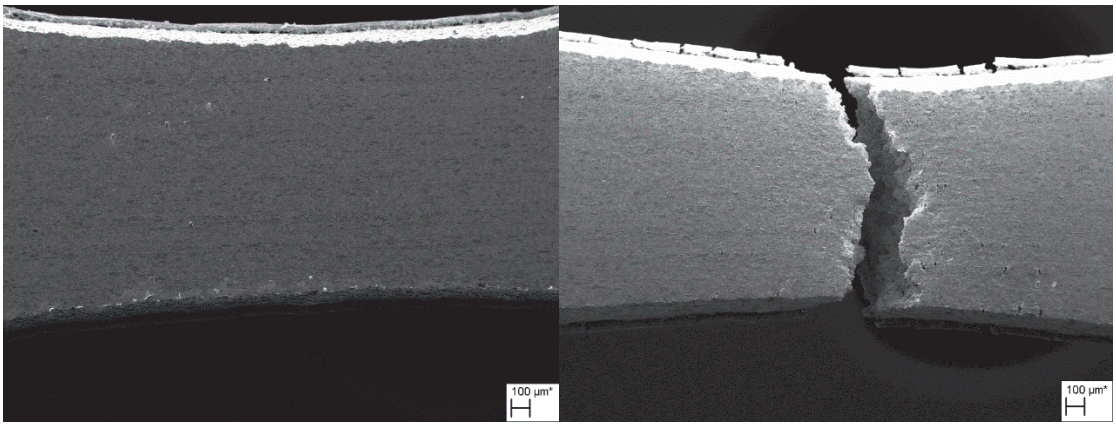


Figure 3 SE image of a round sample (internal label 19436-VS3) before (left) and after (right) the tensile test

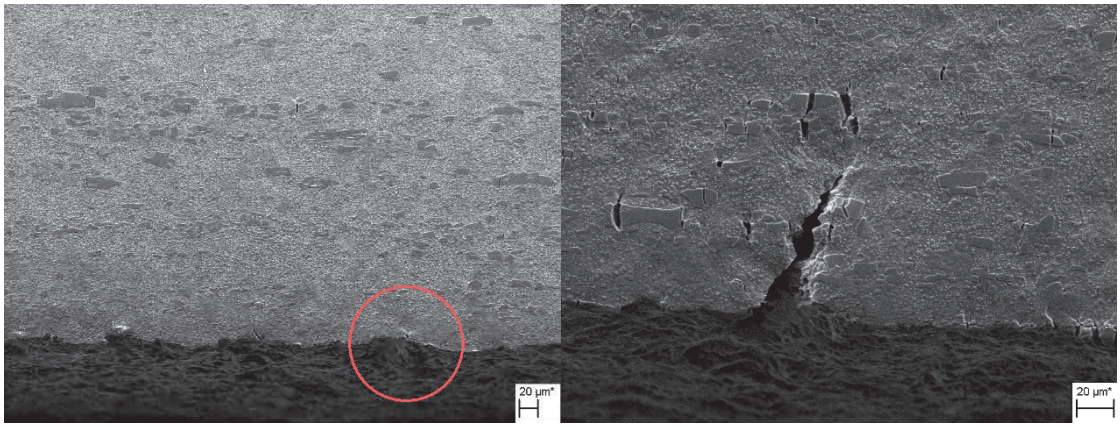
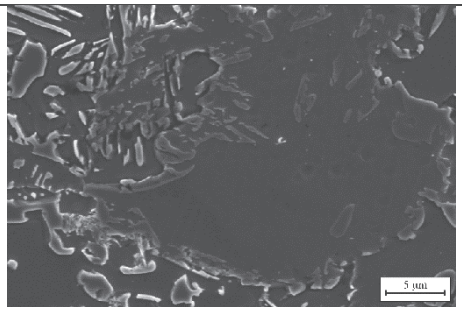
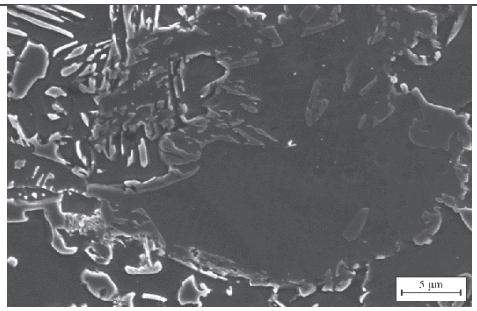
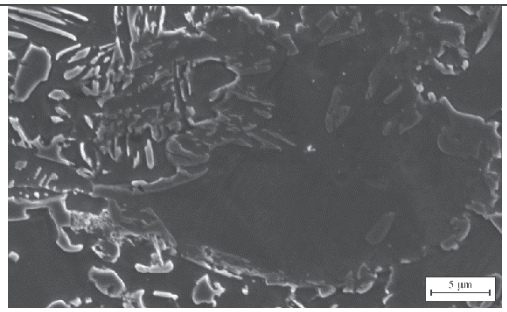
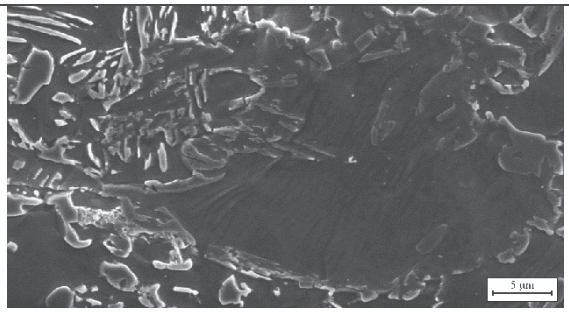
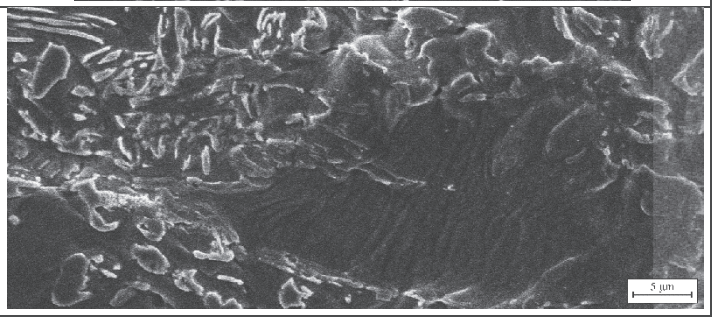


Figure 4 Different stages of crack development (round sample 19436-VS3). Left: initation, right: crack spread during plastic deformation

Table 1 shows the changes in the microstructure by extension of one particular grain during different phases of the tensile test. Elongation (ε) was calculated from the images proportions (image piling with 3 fixed points) and at the end of the test, when the sample breaks is 54.9 %. Δl is absolute elongation of the sample, starting length was 45 mm, F is load. Last two images also display the deformation of the surface, where some “waves” can be seen. For SE it is not such a problem while the sample is perpendicular to the electron beam however some particles from the material can raise up from the surface and disturb the signal by shading or charging.

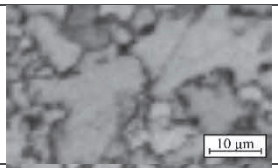
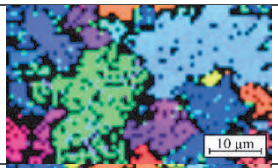
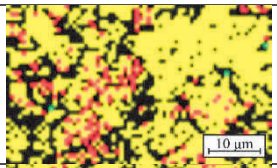
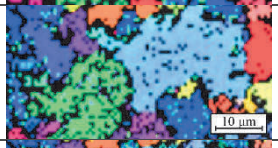
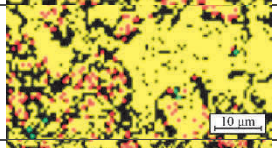
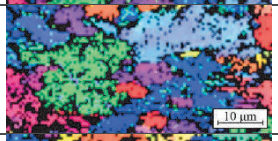
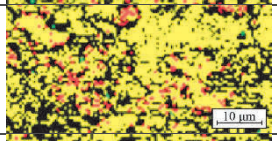
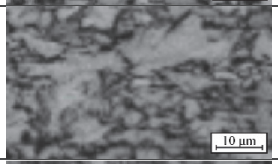
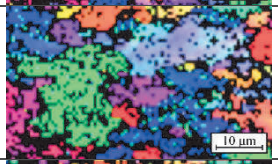
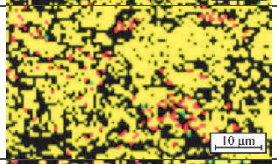
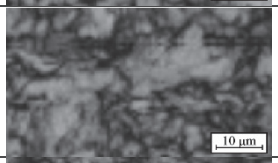
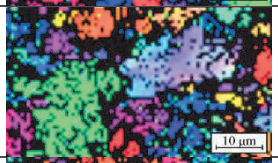
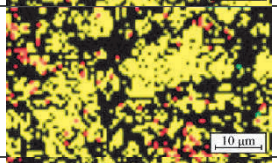
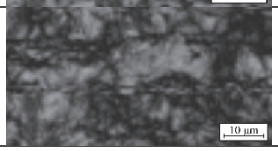
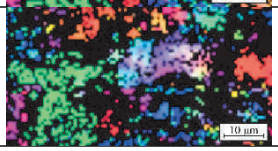
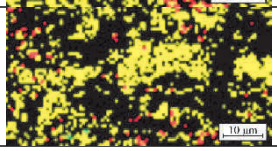
Table 3 SEM images of the same region in continuing deformation

Δl [mm]	F [N]	ε [%]	SEM
0	0	0	
1.00	1 563	3.5	
1.50	2 009	10.3	
2.00	2 212	24.0	
2.5	(break)	54.9	

EBSD mapping requires longer acquisition than SE analysis this means that tensile test needs to be interrupted prior to each mapping. A typical course of a tensile test with EBSD is shown in **Figure 2**. Following table shows not only signal loss for EBSD analysis due to surface deformation but it can be seen how particular grains are

deformed (tensile force works in a horizontal direction) and also one of the ferritic grains (light blue on IPF-Z map) is transformed into two grains.

Table 4 Band contrast, inverse pole figure and phase map derived from EBSD analysis of the microstructure development during tensile test. Images from 6 positions are shown, starting before the experiment (elongation, ε = 0 %, and engineering stress σ_e = 0 MPa)

ε [%]	σ_e [MPa]	Band contrast	IPF-Z	Phase map
0	0			
4.4	314			
9.3	492			
14.4	597			
19.3	664			
29.3	729			

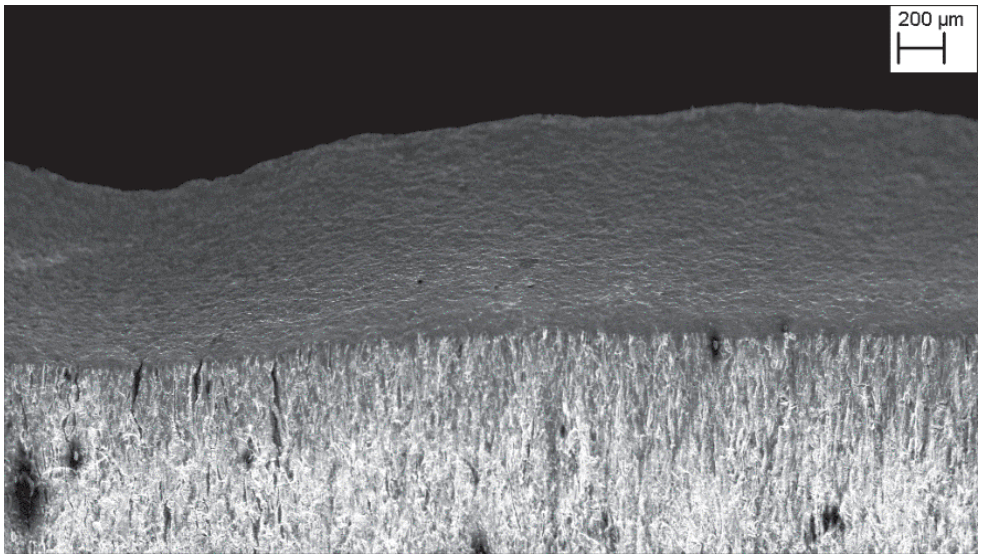


Figure 5 Necking of the sample

Difficulties of in-situ testing are mainly connected with surface deformation, which particularly for EBSD is crucial. Mainly in later phases of plastic deformation, there is significant signal loss of EBSD patterns and growing zero solutions positions due to rippled surface of the sample caused by necking (**Figure 5**). Other situations can occur when there is a high number of inlets in the sample which behave differently than the rest of the material and can pop up. These inlets then form barriers which cause shadow (or have charging effects) on sample surface.

Necking of the sample leads to the loss of alignment with EBSD detector, which is a necessity for EBSD analysis. Several possibilities can offer to minimize this effect none of them is very suitable. When choosing a site for EBSD maps it is difficult to guess total loss of the signal at later phases of the experiment. It is possible to have several examination sites and to analyse all of them at each stage. This however extends acquisition time very much and also it can need a change of settings for EBSD (required for example by shading of the stage on EBSD detector). Other option is to choose site not so close to the region of plastic deformation, this however does not show processes of the main focus. Next, if the structure consist of combination of big grains and small islands of retained austenite, it is likely that austenitic region will be affected by signal loss much more than the grain region. This is also something what is in contrary to the focus when following the austenitic changes in the material. Experimental work is still in process in the focus are possibilities of reducing the signal loss at higher stages of deformation.

4. CONCLUSION

Possibilities of imaging microstructure of steel sample during tensile testing are shown. For experimental work were used samples cut from high strength low-alloyed TRIP steel. All experiments were made using in-situ tensile stage compatible with SEM enabling either SE or EBSD analyses. To obtain good results, preparation of samples must be excellent. Metallographic grinding, polishing and for EBSD observation electrochemical polishing of the surface is required to achieve sufficient EBSD signal. However, deformation experiments cause deformation of the sample and its surface. This causes signal loss and growing numbers of zero solution in the EBSD map.

Imaging at the early stage of deformation is already mastered some challenges in EBSD mapping at high deformation remain. Analysing of the microstructure during tensile testing can provide nice view of changes proceeding in the material and thus give important information besides that acquired only from initial and resulting state.

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