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INVESTIGATION OF HIGH STRENGTH STAINLESS STEEL USING SMALL SPECIMEN TEST TECHNIQUES - TENSILE AND FATIGUE PROPERTIES

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ABSTRACT

The evaluation of mechanical properties of in service components is a critical issue for many technological units in order to estimate safe residual service life. Therefore, there are being used and further developed non-destructive as well as semi-destructive testing methods allowing material properties evaluation of the operated components. Some methods are using empirical correlation allowing of conversion of some measured characteristics into "standardly" used terms (e.g. Small Punch Test, Automated Ball Indentation..). Some other methods focus on miniaturization of standard shape testing samples. Basic properties used mostly for the service life evaluation are tensile strength, impact notch toughness or impact notch toughness transition curve, fracture toughness, creep and high cycle fatigue.

The current paper is dealing mainly with high cycle fatigue properties determination with the use of mini samples that can be extracted out of in service components. Additionally, small size Micro-Tensile Tests are being also applied in order to provide initial stress levels for subsequent high cycle fatigue tests. The material investigated is precipitation hardening steel X1CrNiMoAlTi 12-11-2. Small size specimens are extracted from the experimental block by Electro Discharging sampling machine that is used for a real in-service components sampling in order to keep current experimental program as close to reality as possible. Tensile test properties as well as high cycle properties are assessed with the use of small size specimens and standard size specimens. Very good agreement is found between both specimens sizes proving possibility of small size specimens' application to residual service life assessment with higher accuracy in comparison to the methods using empirical correlations for the properties evaluation.

Keywords: High cycle fatigue, Tensile test, small size samples techniques, precipitation hardening steel, Digital Image Correlation (DIC).

INTRODUCTION

The current paper is going to present potential of small size samples application to mechanical materials properties determination. The aim of these methods application is the residual service life evaluation, evaluation of local material properties and material properties assessment in cases when limited volume of the experimental material is available (e.g. development of nanostructures materials by sever plastic deformation methods, development of special thermo-mechanical treatment with the use of physical simulators…). There has been developed several non-destructive or semi-destructive methods such as Small Punch

Test (SPT) (Konopik, 2012) or Automated Ball Indentation (ABI) (Haggag, 1993) used for considered purposes.

Drawback of these methods is they are using for the properties quantification some correlations with limited validity and higher tolerance bounds stemming from the measurement and evaluation uncertainties and also accounting for different loading modes between these methods and standard testing methods (e.g. SPT x Charpy impact notch toughness, ABI x fracture toughness,...).

Therefore development of small size specimen techniques using miniaturized standard size samples is important, because these tests maintain very important advantage - the same loading mode as standard test samples (Brynk, 2012). Problem with samples miniaturization is mainly availability of appropriate measuring devices suitable to test samples of volume of few cubic millimeters. Load transducers are not so critical, but for example direct strain measurement on mini-tensile sample requires special optical methods, such as Digital Image Correlation (DIC), that are widely available just recently. Using advantage of application of the newest measurement equipment and techniques, miniaturized samples can be successfully used providing much more reliable data than presently used methods using correlation approach. Furthermore, such a data can be used as an input data influence on FEM simulation (Dzugan, 2014) or for calibration of fracture locus (Spaniel, 2014).

The experimental program here is performed on X1CrNiMoAlTi 12-11-2 steel. There are performed standard tensile tests and high cycle fatigue tests as well as small size tests. Small size samples are machined from the experimental material extracted by portable sampling device. The device is used for material extraction from real in service components in order to simulate real component service life assessment, where material has to be extracted without the component destruction. Results obtained are very positive, very good agreement between standard size and small size specimens is found for the material investigated.

EXPERIMENTAL MATERIAL

The experimental material X1CrNiMoAlTi 12-11-2 stainless steel is considered here. The chemical composition of this steel alloy is summarized in Table 1. It is precipitation hardened stainless steel of very high purity. It exhibits excellent mechanical properties in the longitudinal and transverse directions and also excellent balance between strength and toughness properties, and very high fatigue resistance, too.

Table 1 Chemical composition of X1CRNIMOALTI 12-11-2 steel alloy

C	Si	Mn	Cr	Mo	Ni	Al	Ti
<0.002	0.020	0.037	12.635	1.922	9.886	1.465	0.324

Prior to testing the material was heat treated according to the red curve, as shown in Fig. 1. The solution annealing was conducted at 532 °C for 8 hours in air. In accordance with the temperature conditions and Fig. 1, the final UTS should theoretically reach around 1,640 MPa. The surface of semi-finished rod bar was protected by Tinderex paint prior to annealing in order to minimize decarburization. Due to the dimensions of the bar, the heat treatment consists of an extra holding time at 350 °C for 30 minutes. The heating rate was conformed to the diameter of 200 mm and was set at 10 °C per minute. After 8 hours at 532°C, the round bar was quenched in water. The microstructure consists of low-carbon martensite with high concentration of secondary particles. The microstructure obtained is shown in Fig. 2. The particles are randomly distributed in the matrix and located in grains and on grain boundaries.

The average grain size of 9 µm was evaluated according to ASTM E112 standard for the linear intercept method, see Table 2. The grain size evaluation helps to estimate the grain size effect (Heming, 2007, Kundan, 2006).

Table 2 - Results of average grain evaluated according to ASTM E112 standard for the linear intercept method

Grain Size of		**µm**	**G**
X1CrNiMoAlTi 12-11-2	core	8.59	10.0
	edge	9.28	10.0

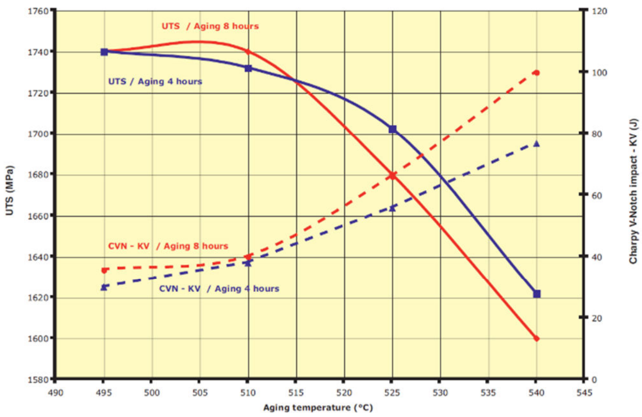


Fig. 1 - Tempering diagram, aging time of 8 hours at 532 °C

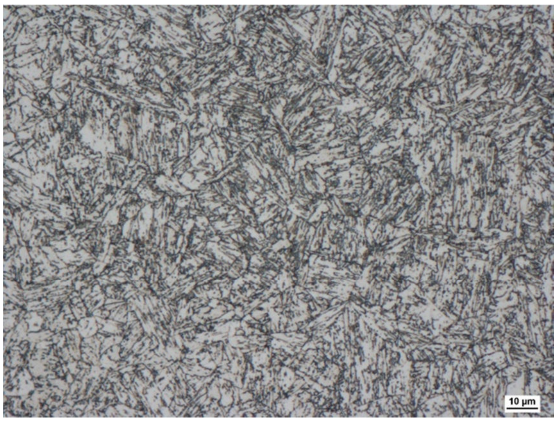


Fig. 2 - Microstructure of X1CRNIMOALTI 12-11-2 steel after heat treatment; 500x magnification

STANDARD TENSILE AND HCF TESTS

As a verification of sub-size specimens test results, the standard tensile and high cycle fatigue tests were carried out on the material investigated. The orientation of the specimens was strictly observed during the sampling and kept the same for all specimens. In order to evaluate properties homogeneity over the investigated work-piece, tensile test specimens were extracted from the surface and also from the central part. In the case of HCF tests, only specimens from the edge "K" were considered. All tests were carried out at room temperature.

Samples for standard tensile tests were cut out from the core and edge of the round bar in the longitudinal direction. There were extracted and tested three specimens from each sampling location. Labeling denotes the sampling location, “S” means core, “K” states for edge. Tests were conducted on servo-hydraulic testing system Inova with a load capacity of 200kN at temperature 20 °C. Tensile test strain rate of $0.0004 s^{-1}$ was applied. The tests were performed according to CSN EN ISO 6892-1, *Metallic materials - Tensile testing - Part 1: Method of test at room temperature*. A longitudinal deformation of a loaded specimen was measured by means of mechanical extensometer with gauge length of 25 mm. Dimensions of the samples were measured prior to the testing and after the test for determination of stress and plastic behavior quantities determination (Young´s modulus - E, Proof stress - YS, Ultimate Tensile Strength - UTS, Uniform Elongation - UA, Elongation - A and Cross Section Reduction - CSR). The average values of tensile test results is summarized in Tab. 3.

Tab. 3 - Average standard tensile test results

Specimen	Temp. °C	E GPa	YS MPa	UTS MPa	UA %	A %	CSR %
Avg. K	20	212,8	1592,1	1648,3	1,0	11,5	61,7
Avg. S	20	211,6	1553,9	1608,1	1,1	11,6	59,8

High Cycle Fatigue (HCF) tests were conducted on the magneto-resonant fatigue testing machine Vibrophore Rumul for loads up to 250 kN in tension-compression mode with load ratio of R=-1. Batch of 10 test-pieces with diameter of 8 mm loaded at various stress levels was tested.

Tests were carried out in load control mode at frequency of about 120 Hz. Specimens were cyclic loaded until failure using a sinusoidal waveform. The amplitude was kept at a constant value throughout the test and the number of loading cycles to failure was indicated. The test control parameters were recorded as well next to standard data to ensure that any peaks in applied load didn't exceed desired load level throughout the test. Each test was terminated at specimen failure or after reaching 10^7 cycles.

Prior to these tests, tensile test were conducted and evaluated. According to the tensile test results, the first loading levels were proposed. In the case of the first specimen, the loading level was set as 2/3 of YS. Further loading levels were chosen according to previous tests at different levels.

The Wöhler curve was compiled on the basis of measured values. The fatigue limit σ_C was evaluated as the highest value at which the sample didn’t crack even after 10^7 cycles.

MATERIAL SAMPLING AND SUB-SIZE SPECIMENS

In the case of sub-size specimens testing, a special procedure was applied to the experimental material extraction, simulating a real process of the material extraction from in service components. All sub-size specimens were subsequently machined out of such an extracted material. The size of specimens was adjusted to fit into the available volume of the experimental material that is possible to extract in semi-destructive manner. Tensile tests and mainly high cycle fatigue tests were carried out on these test-pieces.

A portable electric discharge sampling equipment (EDSE) features easy handling, low-pressure coolant circuit (minimize spatter), quick electrode release and replace and a possibility to design own geometry and last but not least high sampling efficiency. Due to this fact the device is suitable for an in situ sampling out of in-service components, Fig. 3. The device enables to adjust a wide range of work-piece thicknesses whereas the work-piece width of 20 mm is given by the electrode geometry. The thickness of the extracted material can be up to 5 mm. In our case, the depth was set to 4mm using the adjustment screw. Used electrodes were customized in order to reduce a stress concentration on edges and were made of W-Co alloy. Each extraction took approximately 1 hour. Three small materials were extracted. The influence of electro-erosive machining on the work piece is demonstrated in the Fig. 4. The depth of the affected layer appeared to be slightly less than 10 μm thick.

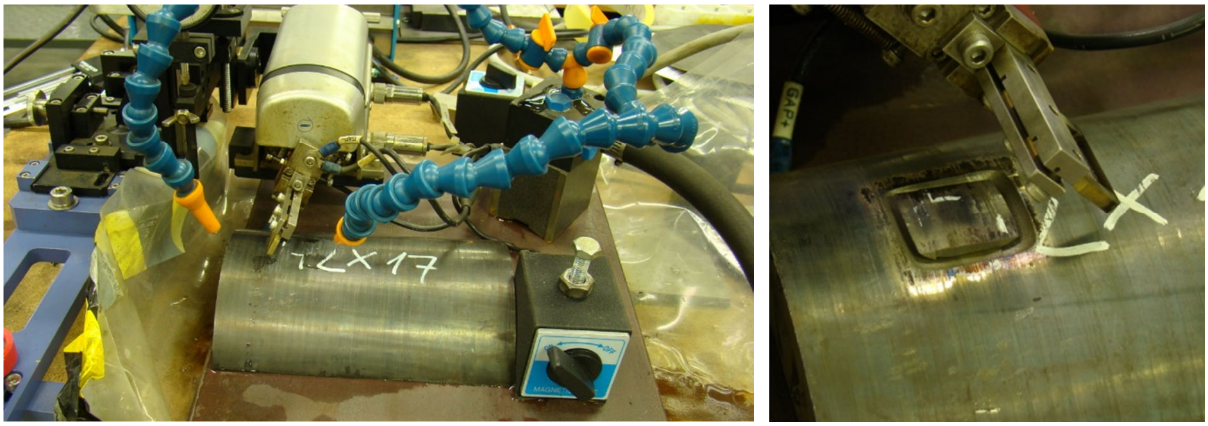


Fig. 3 - The set-up of EDSE; whole device set up on the left and extracted piece detail on the right

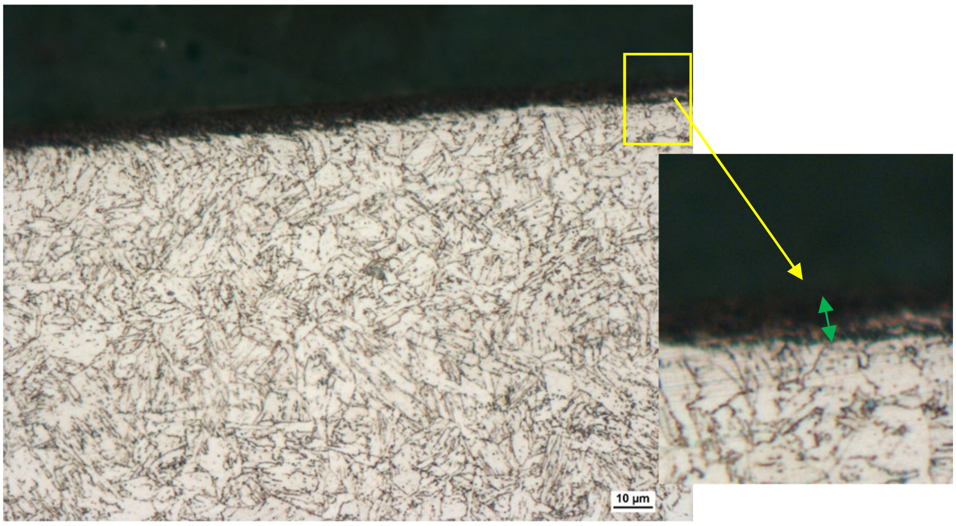


Fig. 4 - Influence of EDS machining on microstructure - affected depth is less than 10 μm

Sub-size specimens' dimensions and geometries were developed on the basis of available extracted piece of the experimental material. The material volume is limited and thus specimens' size and its distribution within the extracted material was carefully plan in order to utilize maximum of the material available for test-pieces. Specimens outline and cutting scheme within the extracted piece of material can be seen in Fig. 5.

Following specimen geometries were proposed for a tensile test (Fig. 6a) and a HCF test (Fig. 6b). Specimens were produced by CNC turning center. A surface roughness of the specimens after CNC turning didn't exceed 1.4 μm, as it was measured with a Surtronic S25 profilometer. Results of the surface roughness measurements can be seen in Fig. 7. In the case of HCF specimens after turning, grinding was applied to reach the average roughness of 0.2μm by means of wet sanding with 320, 600 and 1200 grit silicon carbide sandpapers.

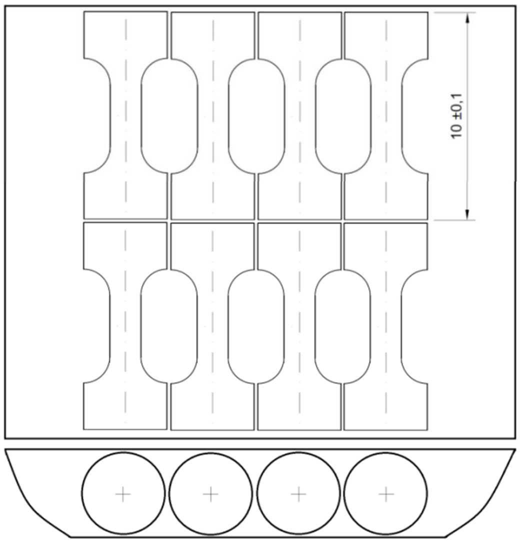


Fig. 5 - Specimens cutting scheme within EDSE extracted material

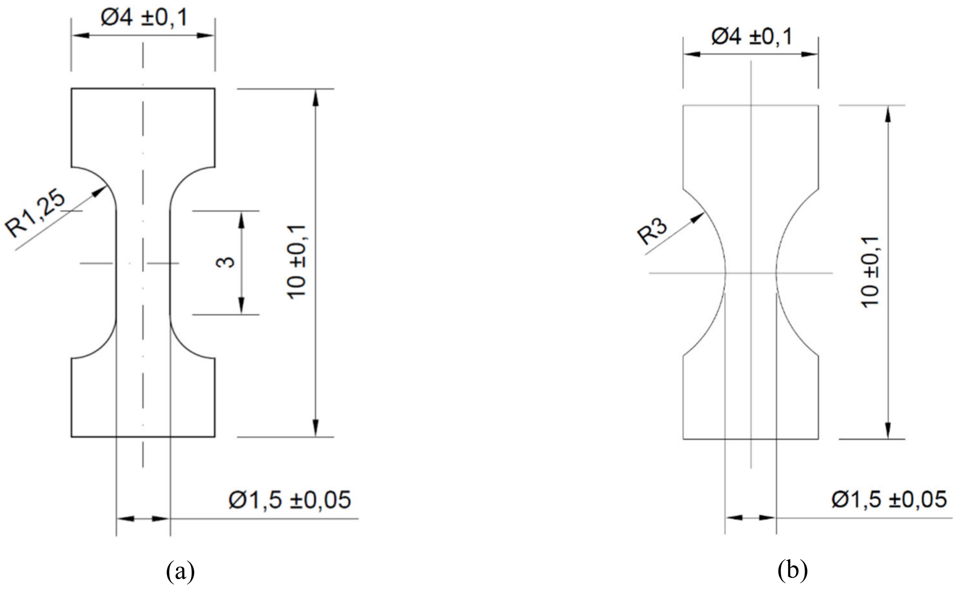


Fig. 6 - The geometry and dimensions of mini tensile (a) and fatigue specimens (b)

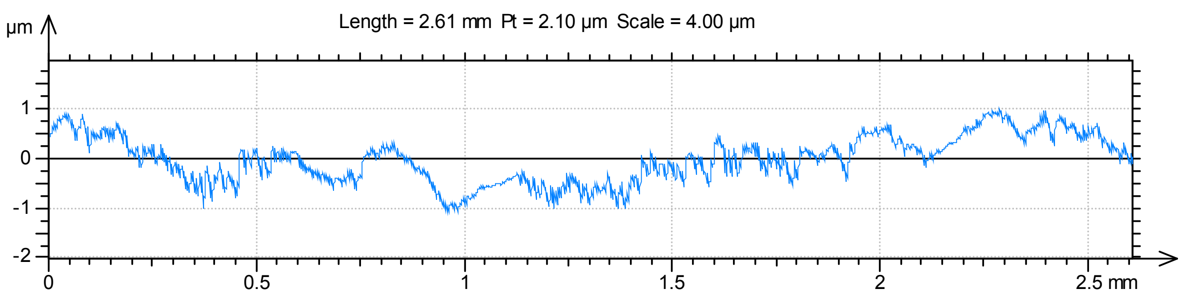


Fig. 7 - A surface roughness tester record

MICRO-TENSILE TEST (M-TT)

M-TTs (Gianola, 2009) were performed at room temperature at the same quasi-static strain rate as in the case of standard samples strain rate of 0.0004 sec^{-1}. Tests were performed with the use of the testing machine using a linear drive with a loading capacity of 10 kN. A tensile load was applied to a specimen until fracture. Strain measurement was done with the use of Digital Image Correlation (DIC) system.

Test samples were taken from a large metal block using the EDSE and furthermore the work-piece was machined with a classical machine tool to reach its final shape in Fig. 6a. After finalization of samples machining, the diameter was measured and recorded. Three micro-tensile tests were prepared. A white coat was applied on the sample surface. Subsequently, black color was sprayed over the white layer in order to create fine, random speckle pattern with high contrast necessary for DIC measurement. The number and size of speckles depends on an area of interest and on a resolution of CCD camera. DIC system enables accurate displacement and strain measurements (Leitão, 2012). A strain distribution is shown in Fig. 8. In this case, DIC system served as an optical strain gauge. Individual images are captured by CCD camera with a particular frame rate in order to describe the sample behavior throughout a test. The calibration of the system is provided by an operator either before or after a batch of tests. Prior to testing, a verification of the alignment of the testing machine was made using alignment fixtures. This eliminates the imposition of bending strains and stresses on specimens under the test. The start of testing machine and the image capturing is at the same time to ensure that both times correspond to each other. At this point, there is no barrier to determine mechanical properties such as E, YS, UTS, UA, A and CSR and construct stress-strain curves. The elongation A_5 was not directly evaluated because of too short gauge length of the sample. Thus the following equation (1) was used for an evaluation of elongation

$$A_x = \frac{UA_m * L0_x + (A_m - AgUA_m) * L0_m}{L0_x} \tag{1}$$

where

A is elongation [-],

UA is uniform elongation [-] and

L0 represents initial gauge length [mm].

Index "**m**" is for originally measured values with considered gauge length (in the current case = 1) and "**x**" is the factor specifying gauge length to which are originally measured values are converted (5 in the current case) (Dzugan, 2010).

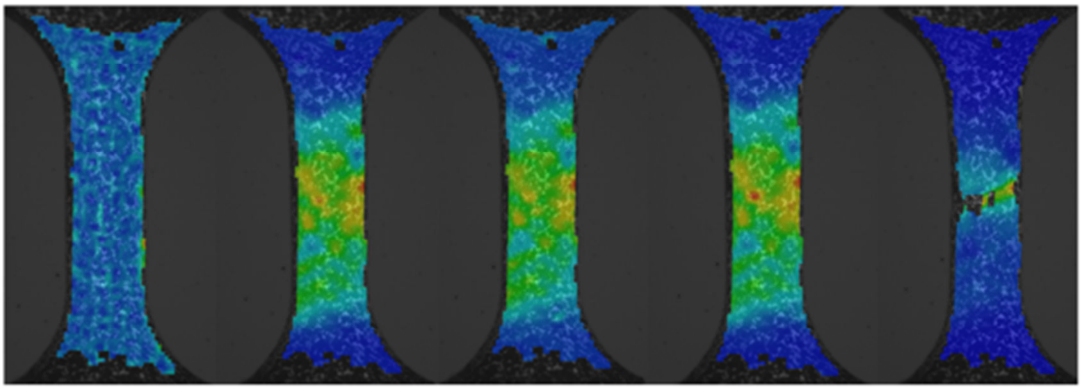


Fig. 8 - Strain measurement by means of DIC on micro-tensile samples

Micro-tensile tests results are summarized in Table 4. Very good agreement between sub-size and standard size specimens can be clearly seen from the Fig. 9, which is in agreement with previous findings in (Džugan, 2014). The average mechanical properties are following: E = 214 GPa, YS = 1593 MPa and UTS = 1646 MPa. As clearly seen from table 3, there is not a huge scatter between standard and sub-size tensile test results. The crack occurred within the specimen gauge length and a significant necking process was observed during the test. Mechanical properties were reached without any numerical correlations except for an elongation (A5). The elongation was computed based on equation (1).

Table 4 - Results of micro-tensile tests and comparison with standard tensile tests

Specimen	Temp. °C	D_0 mm	D_u mm	E GPa	$Rp_{0,2}$ MPa	Rm MPa	Ag %	A_5 %	Z %
MT1	20	1.50	0.92	211.8	1594.9	1640.8	1.2	10.9	62.4
MT2	20	1.50	0.91	215.8	1592.3	1650.6	1.4	10.9	63.2
MT3	20	1.50	0.92	212.4	1593.2	1645.5	1.4	10.9	62.8

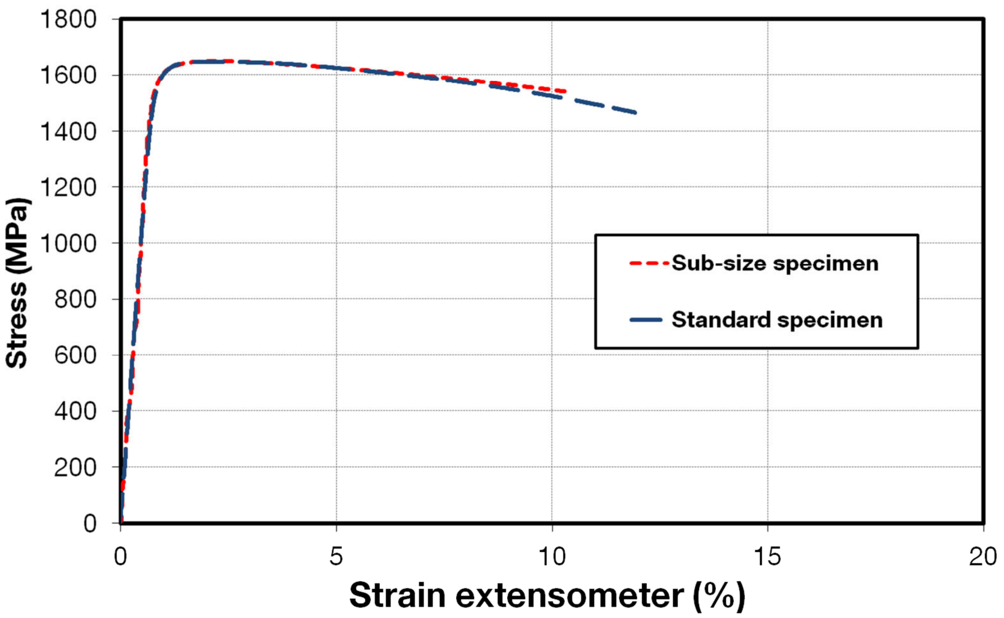


Fig. 9 - A micro- tensile test record in comparison with a standard tensile test record

SUB-SIZE HIGH FATIGUE TEST

HCF tests on miniaturized specimens were conducted on the resonant fatigue testing machine Vibrophore Rumul for loads up to 20 kN in tension-compression mode and on servo-hydraulic testing machine MTS Bionix with load-carrying capacity of 25 kN. Tests were carried out in load control with load ratio of R=-1 at frequency of about 50 Hz. Dimensions of samples were measured using an optical measuring system to avoid scratching. If a scratch occurred the surface, it was additionally regrinded and measured. Wedge grips are attached to the testing machine in appropriate way to avoid bending stresses that was done with the use of alignment fixtures. After this, a fatigue specimen was put in jaws of the testing machine and the pre-load was set to zero. All tests were conducted at room temperature. According to the micro-tensile test results, the first loading levels were applied. In the case of the first specimen, the loading level was set as 2/3 of 1590 MPa (YS) as in the case of a standard HCF. Each test was performed until failure occurred or after reaching 10^7 cycles. Size comparison with a standard HFC specimen can be seen in Fig. 10.

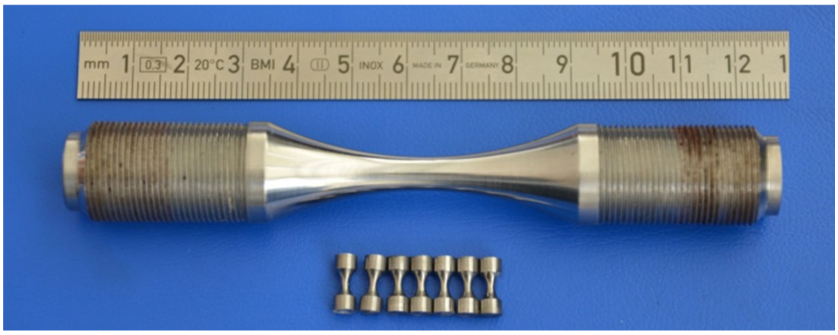


Fig. 10 - Comparison of HCF specimens

Obtained results from sub-size and standard fatigue tests are shown in Fig. 11. This test results are presented as the number of cycles to failure which are plotted against the stress amplitude in the semi-logarithmic scale. Red dots represent sub-size HCF tests and the green line denotes its fatigue limit. Those of specimens which survived 10^7 cycles are denoted by arrows. Sub-size fatigue test results exhibit higher scatter and this corresponds to fit correlation coefficient R^2 of 0.66. This fact shows a worse fit of linear regression in comparison to the standard one. This is expected feature and it can be caused by several factors such as an inhomogeneity of steel which is represented by secondary particles in the matter, notch sensitivity, or surface conditions. The summary of fatigue limits is shown in Table 5. The results obtained with the use of both specimen batches yield very similar results that is very positive result.

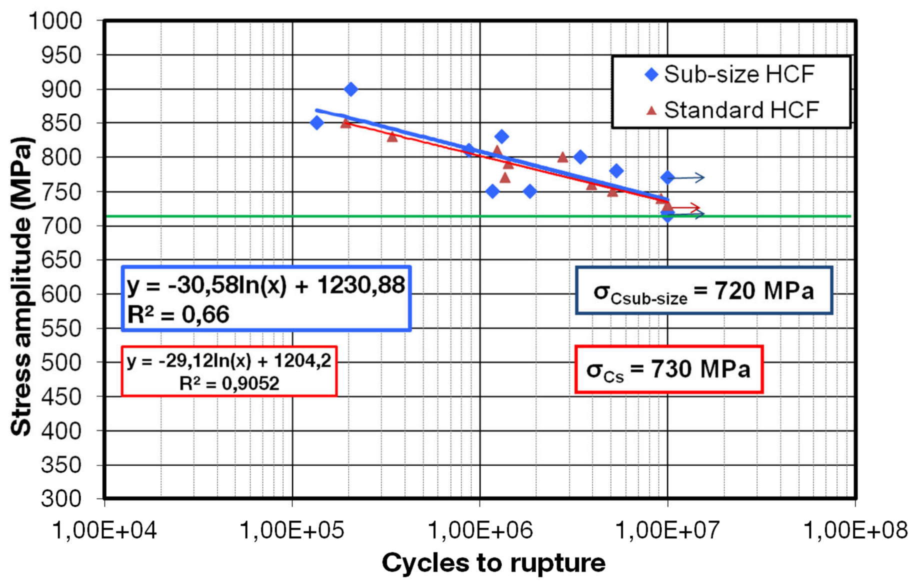
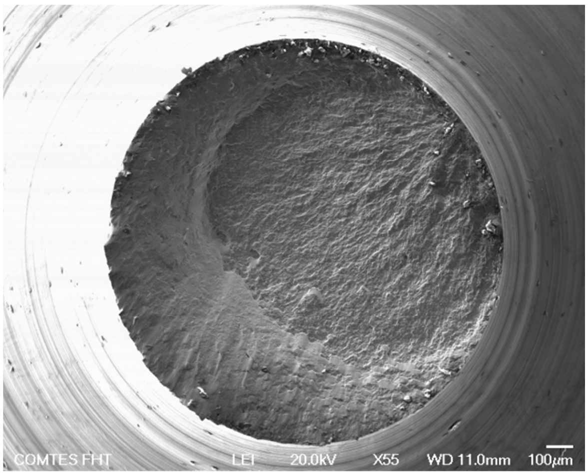


Fig. 11 - S-N curve of X1CrNiMoAlTi 12-11-2 steel for Sub-size and Standard HCF tests

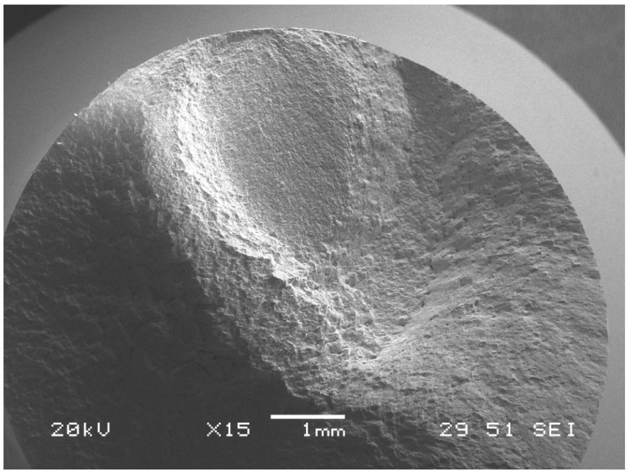
Table 5 - Results of standard and sub-size HCF tests

Type of test	Fatigue limit [MPa]
Non-standard HCF	720
Standard HCF	730

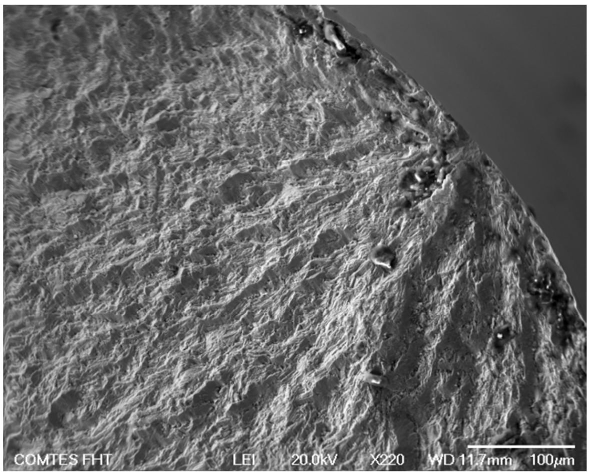
After fatigue tests, the fracture surface morphology of selected specimens was examined with the aid of a JEOL JSM 6380. The fracture surface morphology of sub-size specimen shows a good agreement in comparison with standard ones. On the fracture surfaces, the fine striation and the fracture initiation were observed, see Fig. 12. Mostly, the fracture initiation was caused by randomly spread particles which act as a fatigue crack initiator.



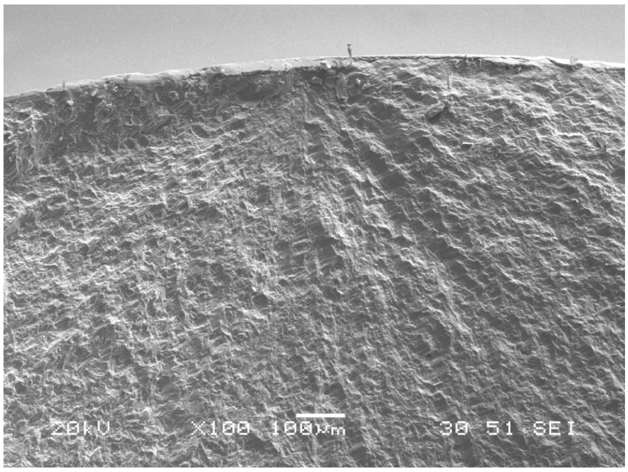
(a) Sub-size HCF at 55x magnification



(c) Standard HCF at 15x magnification



(b) Sub-size HCF at 220x magnification



(d) Standard HCF at 100x magnification

Fig. 12 - Fracture surface morphology of broken high cycle fatigue samples

DISCUSSION

The effect of electro-erosion machining on the microstructure of a MLX17 high strength stainless steel is evident in the Fig. 4, but on the other hand, in this case, the influence of 10 μm thick layer is negligible because this layer is removed during subsequent machining. Despite this fact the erosion influence could have an influence on an in-service component, but this is beyond the scope of this work (Mower, 2014).

A few well known effects of EDSE on surface of in-service components and their minimizations:

- Electrode geometry - optimal electrode designs could reduce the effect of a cutting which might cause, in certain circumstances, as a fatigue crack initiator.

- Structure degradation - for these reasons, it’s recommended to grind off a thin degraded layer on the component surface. Corrosion products may cause an early rupture of the component.

The obtained stress - strain curves and results of sub-size tensile tests are fully comparable with the results of the standard ones. The crucial factor for determining of yield strength is the elastic modulus. Its determination was carried out using DIC system which employs the ARAMIS system and very good agreement with typical values for this kind of the material is found, even for this kind of very sensitive measurement. Due to the specimen geometry, short gauge length mainly, it is not possible evaluate elongation A5 directly. It had to be (re)calculated based on the experimental equation (1). This enabled to compensate a short specimen gage length. Resulting values of the evaluated elongation are also in very good agreement with the results obtained for standard size specimens. Even in the case of cross section reduction evaluation, very close values were attained for both specimen geometries.

HCF Testing of smaller samples usually leads to a higher data scatter that is partly visible on the results obtained. However, the data scatter for sub-size specimens is very reasonable and fitted trends for both data sets overlap each other and the data obtained clearly belong to the same population. The slope of the regression line shows a similar trend and the intercept at 107 cycles is almost identical (difference of 2%). In accordance with this, sub-size tests give very promising results, as it is shown in Table 4. In order to obtain statistically more reliable data, stair case method can be applied. However, in case of real tests, there would be hardly available sufficient material for extensive stair case testing. The results obtained provided very good agreement between sub-size and standard size test pieces, thus the specimens dimensions shown here is suitable for high cycle fatigue material behavior assessment.

CONCLUSION

The presented work is dealing with mechanical properties evaluation of X1CrNiMoAlTi 12-11-2 steel that is used for high loaded components such as parts for aerospace, high pressure pumps or offshore applications. There are high demands on a safe service life of this kind of components and thus possibility of evaluation of actual material properties after several years of use is essential. Therefore some non-destructive or semi destructive method has to be applied in order to assess actual properties and out of that stemming residual service life. There are used small size specimen testing methods such as Small Punch Test, Automated Ball Indentation or some others using correlation between measured terms and terms used for mechanical properties quantification such as tensile strength, yield stress and so on. Big advantage of those methods is its simplicity. On the other hand its big disadvantage is uncertainty of the correlation approach used for the material considered.

The work presented here shows results of high cycle fatigue tests and tensile test with the use of sub-size specimens loaded in the same manner as standard size specimens. This leads to significantly higher accuracy of the data measured with this kind of approach. Main reason why there is a higher accuracy is that there is not converted different loading mode from one test geometry to the other one. Then, the only unknown in the evaluation remains the size effect on considered tests. When this size effect is quantified by FEM simulation or experimentally or by both methods simultaneously, a reliable size conversion factor can be obtained, if necessary. The current results clearly shown that with the use of similar material volume as in the case of e.g. Small Punch Test, there can be machined miniaturized tensile and high cycle fatigue specimens that yield fully comparable results with standard size specimens.

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