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DETERMINATION OF FLOW STRESS CURVES OF ZIRCONIUM ALLOY BY DYNAMIC MICRO-TENSILE TEST USING STRAIN GAUGE TECHNIQUE

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ABSTRACT

One of the main uses of zirconium alloys is in nuclear technology, such as cladding of fuel rods in nuclear reactors, especially water reactors. Due to increasing safety demand of nuclear reactors, crashworthiness has become more important and potential crash accidents are calculated using the Finite Elements Analysis. The present work is focused on the investigation of an experimental zirconium alloy under various strain rates at room temperature. As there is in many cases lack of zirconium alloys for testing, miniature specimens were used. This technique was called micro-tensile test. Further advantage of M-TT is short parallel length (less than 3 mm) and strain rates reachable by these sample with the combination of impact tester IM30T are up to 2500 s^{-1}. Force during a test was measured by strain gauge and the piezo electric load cell. For precise strain measurement, pictures captured by the high-speed camera were evaluated by the ARAMIS system using the Digital Image Correlation method (DIC). Flow stress curves as precise input for FEM simulation determined by micro-tensile test are presented here.

Keywords: Zirconium alloy, high strain rates, dynamic micro-tensile tests, strain gauge, high speed camera, digital image correlation (DIC)

INTRODUCTION

As precise material data of zirconium alloys are unavailable for high strain rates, an experimental zirconium alloy used for nuclear components has been investigated since 2012. In the work (Maresova, 2013), high strain rate sensitivity was measured for zirconium specimens with notches of radii R = 5 mm, 10 mm and 15 mm. In addition, a new testing method for tensile properties determination requiring very little experimental material was developed. This technique was called the micro-tensile test (M-TT).

The developed M-TT has some special features: due to a very small tested volume, it is possible to measure local mechanical properties (Džugan, 2015), anisotropy of thin tubes, flow stress curves at various temperatures (Dzugan, 2014) and also dynamic tensile properties (Konopík, 2014, Rund 2015)). The samples can be obtained from the same material volume as small punch test, but maintaining the same loading conditions as a standard tensile samples, do not need any kind of correlations, enabling direct standard test parameters determination. The M-TT thanks to miniature specimen size allow to attain high strain rates with the use of relatively slow loading velocities in comparison to standard dynamic tests. Moreover, slower loading velocities cause fewer force oscillations typical for high strain rate tests.

MICRO-TENSILE TEST DEVELOPMENT

One of the well-known miniature testing techniques is the Small Punch Test (SPT). The SPT method uses small test disc shaped samples of usual dimensions of 8 mm in diameter and thickness of 0.5 mm. The testing itself is a penetration of a hard ball through the tested sample while the force and ball displacement are measured either at room temperature or non-ambient temperature. The SPT is usually based on conversion of the obtained results into conventional mechanical characteristics. However, it requires known correlation parameters determined for the specific material and must be verified for each new material. Moreover, these correlations have limited validity and higher tolerance bounds stemming from the measurement and evaluation uncertainties due to different loading modes between these methods and standard testing methods (Konopík, 2012). Therefore, the development of small size specimen techniques using miniaturized standard size samples is important because these tests maintain a very important advantage - the same loading mode as standard test samples. The first suggested M-TT geometry was based on SPT specimen to prove that tensile test is possible to perform with the same amount of experimental material. FEM simulation confirmed the same loading and other condition as in the case of standard tensile test (see Fig. 1).

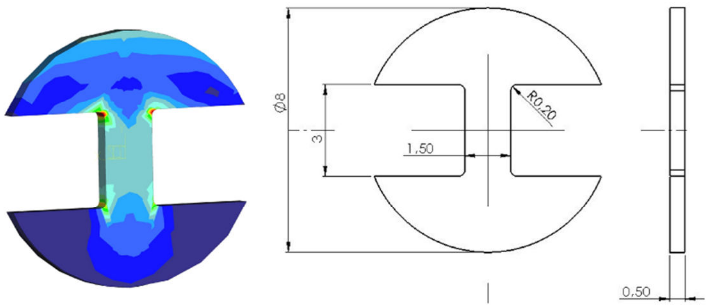

Fig 1 - FEM simulation of M-TT specimen (right), M-TT geometry (left)

In the case of this M-TT sample geometry, a shorter gauge length is used and thus elongation cannot be evaluated in the standard way. The elongation is evaluated with the use of following formula (Dzugan, 2010):

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

Where index m means gauge length used for the test evaluation and x is gauge length into which results are being converted.

The main problem with samples miniaturization is the availability of appropriate measuring devices suitable for testing samples of the volume of a few cubic millimeters. Load transducers are not so critical, but for example direct strain measurement on mini-tensile specimens requires special measuring techniques. Strain measurement on a standard tensile specimen is usually done by a contact extensometer but in case of a very small specimen, it is almost impossible to attach this equipment on the parallel length of the specimen. Considering various strain rates up to the dynamic condition, traditionally used mechanical extensometers could not be used at all. Firstly, the data acquisition rate of the mechanical extensometer is not sufficient during the dynamic event, and secondly, the dynamic test could be destructive to the extensometer itself. Therefore, the strain capturing based on optical measurement was the only option.

There are various methods of optical measurement available for strain measurement - for example laser extensometers or video-extensometers. Latest development in the field of deformation measurements are methods that calculate and evaluate deformation on the whole recorded surface of the specimen - full field deformation measurements. One of these methods is the Digital Image Correlation (DIC) method. The principle of the DIC method has been known since 1970s ((Keating, 1975), (Sutton, 2009) and many others). It is based on the recognition of change in the sequence of images. Stochastic pattern is applied on the surface of the specimen prior testing. The test itself is recorded by one (2D in-plane deflection measurement) or two (3D) cameras. Under the load, the specimen is deformed and so is the applied pattern. Comparing the images, changes in the pattern are registered and displacements and strains are calculated. Systems based on this method enable 3D strains measurements of either testing samples or real components. The color maps in Fig 2 show the distribution of major strain (strain in the direction of the highest achieved strain) over the sample.

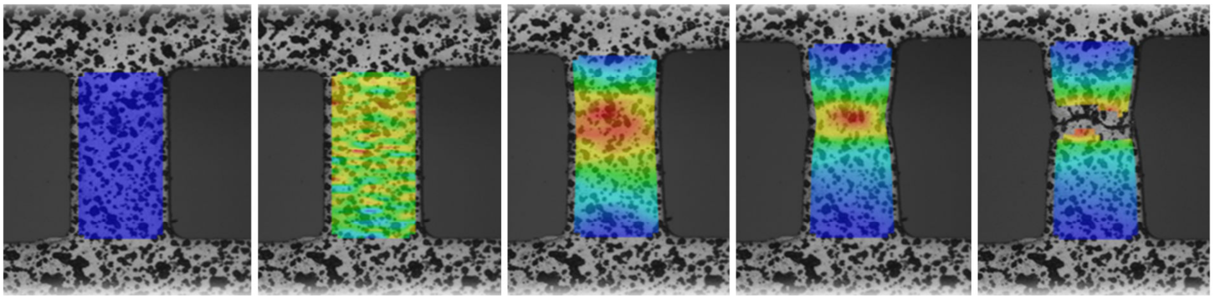

Fig. 2 - Color map of major strain of M-TT sample

The testing procedure was verified for several materials exhibiting a wide range of strength levels. Namely, Al-alloy, Ni-alloy, Titanium Gr. 5, and several steels were compared while tested on standard size specimens and with the use of M-TT. Standard size samples were round with diameter ranging from 5 to 10 mm and in one case steel, segment of pipe was tested. The resulting tensile curves of these tests are graphically summarized in Fig 3. There can be found excellent agreement for all materials investigated between standard size specimens and M-TTs for whole range of strength levels from about 250MPa up to 1250 MPa.

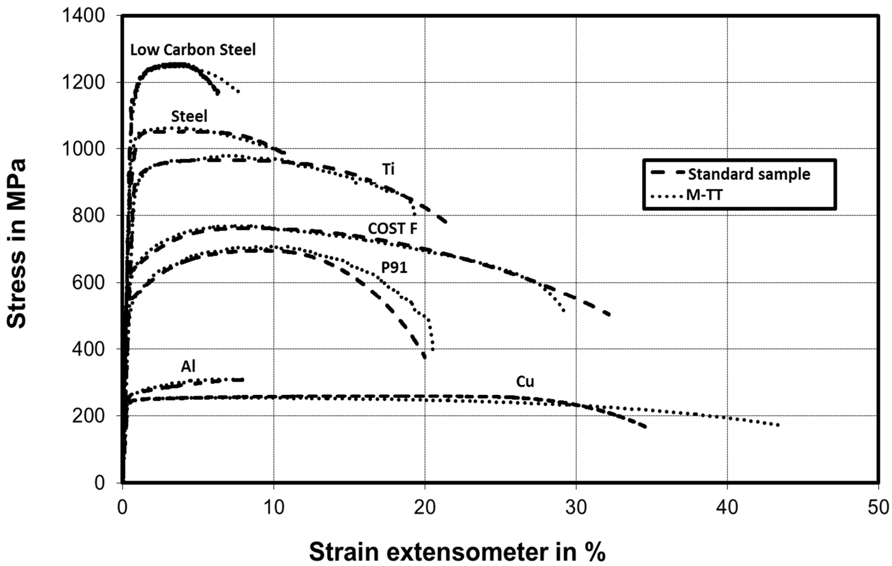

Fig 3 - Comparison of records obtained with the use of M-TT and standard tensile specimen for various metallic materials (Džugan, 2015)

EXPERIMENTAL MATERIAL

Polycrystalline zirconium (of the trade name Zircadine^{TM} Zirconium 702), delivered as a sheet with 1 mm in thickness, was subjected to high strain rate testing. Table 1 shows the chemical composition of the material provided by the manufacturer. Metallographic investigation was performed and an ultrafine-grain structure was observed, see Fig. 4.

Table 2 - Chemical composition of the as-received material- Zirconium 702 (weight%)

Element	**TOP**	**MIDDLE**	**BOTTOM**
C	0.01	0.01	0.01
FeCr	0.08	0.08	0.08
H	<0.0003	<0.0003	<0.0003
Hf	1.0	1.0	1.1
N	0.005	0.005	0.005
O	0.14	0.14	0.15
Zr+Hf	>99.2	>99.2	>99.2

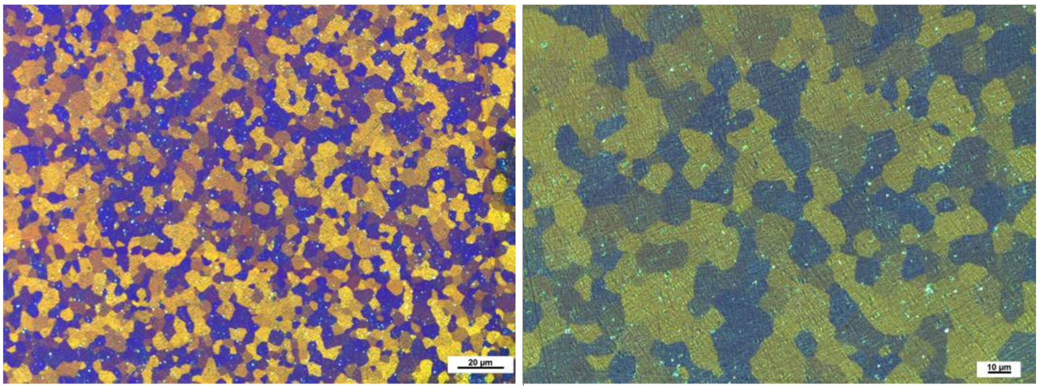

Fig. 4 - Microstructure of Zirconium 702 200x (left) and 500x (right) magnification

To determine the influence of the sample size on mechanical properties, an average grain size evaluation was executed. The average grain size was evaluated according to ASTM E112 standard for the linear intercept method and the results are summarized in Table 2.

Table 2 - Average grain size evaluated according to ASTM E112 standard for the linear intercept method

Grain Size of	µm	G
Zirconium 702	4.75	12.0

It is known that in order to reduce the scatter in measurements, not only the dimensional control but also the ratio between thickness and the grain size is very important. The following guideline is to be followed (Kumar, 2006):

Thickness $\geq$ 10 x Grain size

Due to very fine grain, a minimum difference can be assumed between the results obtained from standard and micro-tensile tests.

EXPERIMENT

Tensile tests were performed at room temperature under different strain rates. The strain rate can be roughly calculated by the velocity of the actuator and the parallel length of the specimen, i.e.

$$\dot{\varepsilon} = \frac{V}{L} \tag{2}$$

where $\dot{\varepsilon}$ is the engineering strain rate, V is the velocity of the actuator and L is the initial parallel length of the specimen, which is the reduced section in the specimen with a constant width.

Thus, the maximum engineering strain rate achievable for a system can be calculated as,

$$\dot{\varepsilon}_{max} = \frac{V_{max}}{L_{min}} \tag{3}$$

where $\dot{\varepsilon}_{max}$ is the maximum speed of the actuator that is limited by the machine capability. L_{min} is the minimum initial parallel length of the specimen, which is controlled by the requirement to achieve the uniaxial stress condition throughout the parallel length of the specimen. The smaller the parallel length, the higher the maximum engineering strain rate is. Therefore, the newly developed M-TT method is convenient to achieve higher strain rates.

The standard geometry used for the impact tester IM30T is a flat specimen with 1 mm in thickness, 10 mm in width and 20 mm in parallel thickness. The modified M-TT specimen geometry for dynamic testing are depicted in Fig 4.

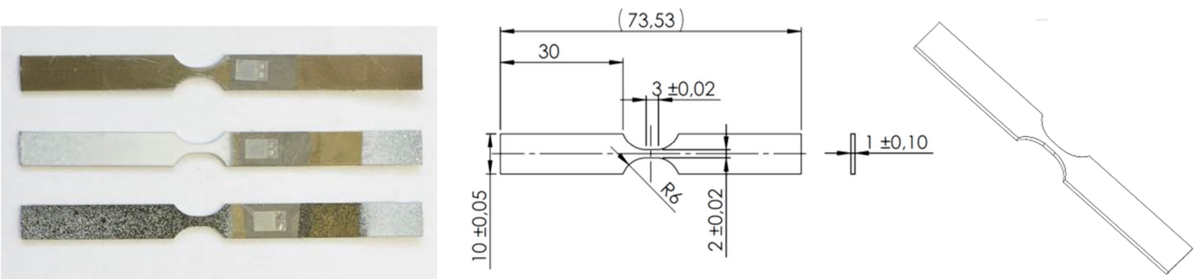

Fig. 5 - Left) M-TT specimen geometry with mounted strain gauge, right) modified M-TT specimen geometry

Quasi-static tensile tests were performed according to CSN EN ISO 6892-1. Currently, high strain rate testing is not widely standardized; however, several documents with recommended procedures exist even for sheet tensile testing (one of them is for example (Opbroek, 2005), but a number of others can be found). Prior to testing, specimen dimensions were measured and an original gauge length for the elongation determination was marked on each specimen. After the test, yield stress was determined as well as tensile strength. The final gauge length was also measured after the test and elongation after fracture was evaluated.

Both the servo-hydraulic system MTS BIONIX with the loading up to 1000 $mm \cdot s^{-1}$ and the impact tester IM30T with impact velocity from 1000 to 7500 $mm \cdot s^{-1}$ were used. In total, 52 tensile tests were performed. Reachable strain rates for specific combinations of the testing machine and the specimen geometry are calculated in Table 3.

The test set-up for MTS BIONIX can be seen in Fig. 6 and for impact tester IM30T in Fig. 7.

Table 3 - Reachable strain rates

Testing Machine	V_{min}	V_{max}	L	$\dot{\varepsilon}_{min}$	$\dot{\varepsilon}_{max}$
	(mm/s)	(mm/s)	(mm)	1/s	1/s
MTS, Standard sample	0	1000	15,0	0	66
MTS, M-TT	0	1124	3,0	0	375
IM30T, Standard sample	1000	7500	20,0	50	375
IM30T, M-TT	1000	7500	3,0	333	2500

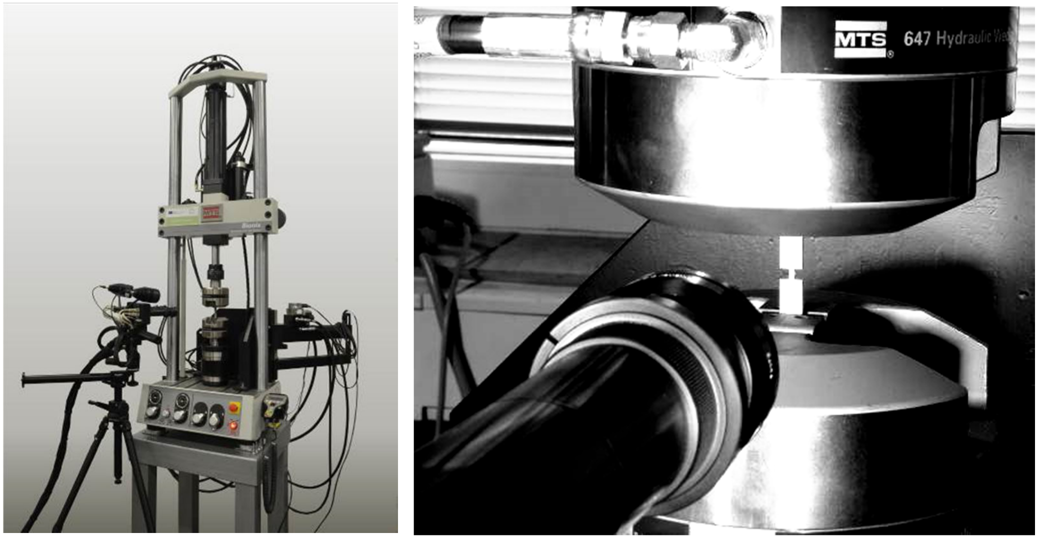

Fig. 6 - Left) MTS BIONIX system, right) detail of strain measuring by ARAMIS

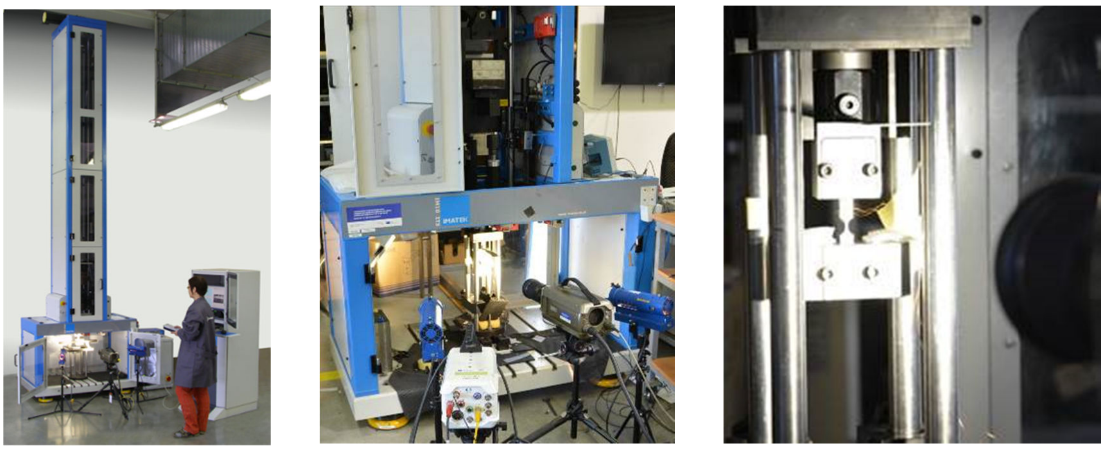
Fig. 7 - Left) impact tester IM30T, middle) test setup with two high speed camera, right) detail of griped specimen with strain gauge for precise force measurement

During the dynamic micro-tensile test, one high speed camera Phantom v711 was used for capturing of the specimen parallel length for further strain evaluation and the second high speed camera monitored the whole setup for further evaluation of unexpected phenomena (bad alignment, lateral oscillation etc.). The recorded images were then uploaded to the evaluation software ARAMIS and calibrated (based on the initial width of samples). Due to very fast loading, maximal speed of 680,000 fps at reduced resolution of 128 x 8 was used and therefore it was not possible to calculate strain distribution as precise as in the case of small samples under quasi-static condition (Fig. 1). However, strain development was possible to measure precisely.

The screenshots from these cameras are shown in Fig. 8 and Fig. 9 and major strain from ARAMIS calculation of the M-TT under dynamic condition is depicted in Fig. 10.

Fig. 8 - High speed camera screenshots -

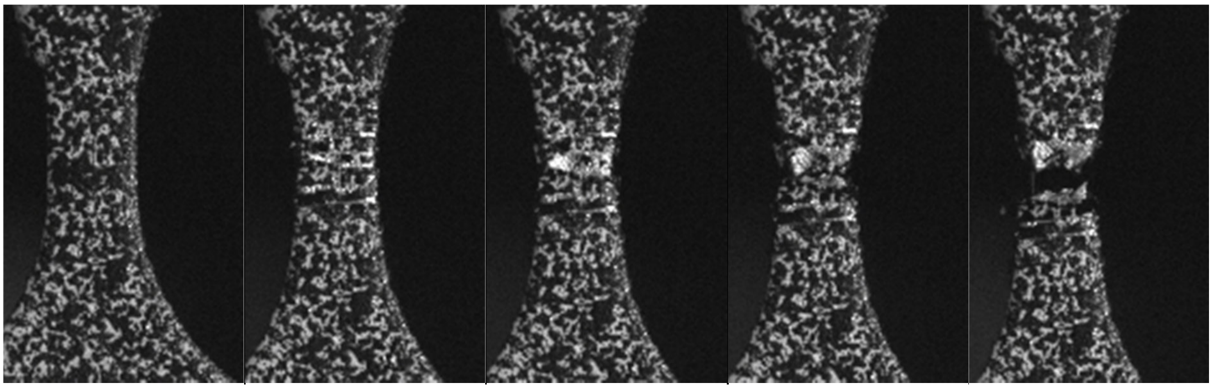

Fig. 9 High speed camera screenshots -

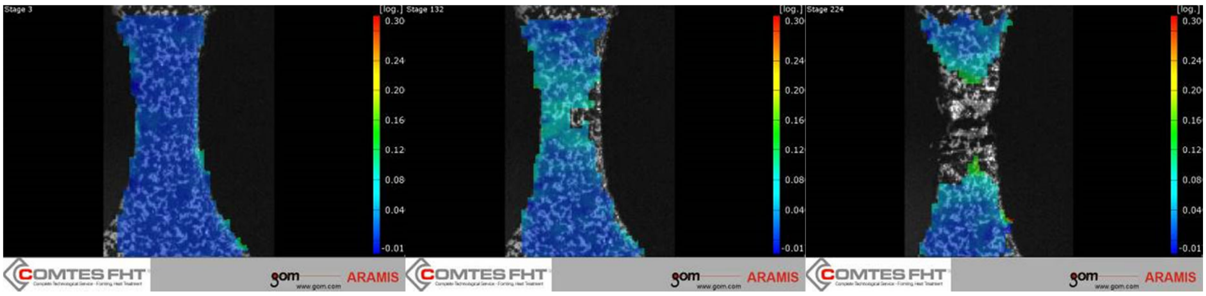

Fig. 10 ARAMIS major strain of the M-TT under dynamic condition

RESULTS AND DISCUSSION

Using the standard tensile samples, the strain rate up to 66 s^{-1} could be reached on the servo-hydraulic system MTS BIONIX (M/Standard). For a higher strain rates, the impact tester IM30T had to be used (D/Standard). Using the M-TT (M/M-TT), strain rates from 0.0015 s^{-1} to 375 s^{-1} were reached with the use of the servo-hydraulic system MTS BIONIX and one type of geometry only. Moreover, improvement M-TT force measurement using strain gauges (which were attached on both surfaces of the specimen) enables to use impact tester IM30T and it makes reachable strain rate of 2500 s^{-1}.

Results from standard tensile samples were compared with M-TT results at the corresponding strain rate (see the comparison of UTS in Fig.11 as an example) and it was found very good agreement. Therefore, only the obtained force-displacement records from M-TT were used for flow stress curves creation, see Fig 12.

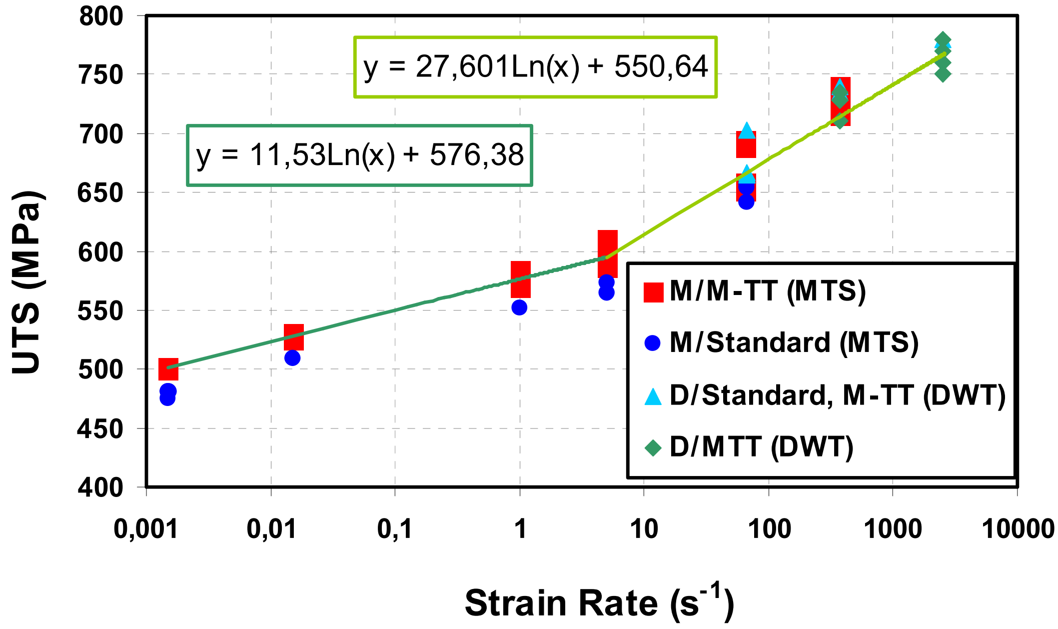

Fig. 11 - UTS dependence on strain rate

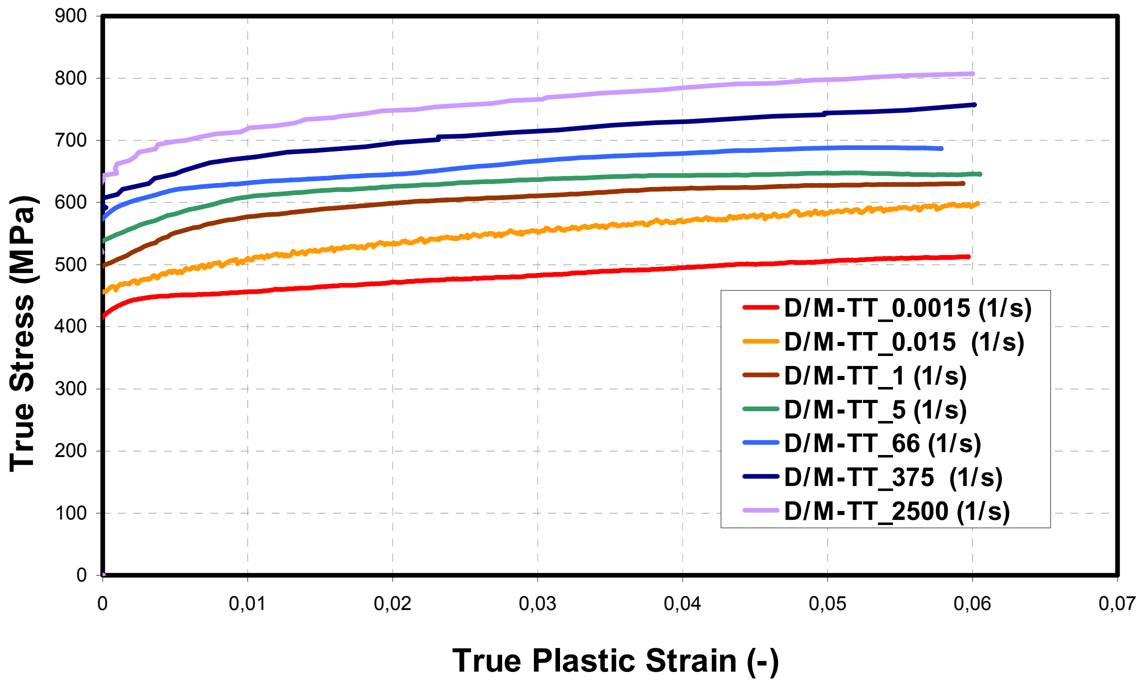

Fig. 12 - Flow stress curve obtained using M-TT performed on MTS and DWT

CONCLUSIONS

Within this work, tensile tests under quasi-static and dynamic conditions using standard and M-TT specimens were performed. The investigated material was zirconium sheet with 1mm in thickness. Precise strain measurement is the main problem of both high strain rate and small tensile specimens testing. Therefore, the high speed camera and the ARAMIS system were used for the evaluation of the precise strain and this solution was suitable for both cases. In total, 52 tensile tests were performed.

Tests were carried out at room temperature with initial strain rates ranging from 0.0015 s^{-1} to 375 s^{-1}. Using the standard tensile samples, the strain rate up to 66 s^{-1} could be reached on the servo-hydraulic system MTS BIONIX and for a higher strain rates, the impact tester IM30T had to be used. However, using the M-TT samples, strain rates from 0.0015 s^{-1} to 375 s^{-1} were reached with the use of the servo-hydraulic system MTS BIONIX only. Moreover, improvement M-TT force measurement using strain gauges enables to use impact tester IM30T and therefore, the reachable strain rate was up to 2500 s^{-1}. The experiment also proved the usability of the M-TT for dynamic properties measurement.

The combination of the DIC system and high strain rate testing promises the possibility to ensure highly precise input data for computer modelling and material behavior prediction.

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REFERENCES

[1]-Dzugan, J.; Novy, Z.; Konopik, P.; Improvement Of Fatigue Properties Of 34CrNiMo6 Steel By Controlled Thermomechanical Treatment, METAL 2010: 19th International Metallurgical And Materials Conference, May 18-20, 2010, Roznov Pod Radhostem, Czech Republic. pp. 421-426.

[2]-Dzugan, J, Zemko, M. Input data influence on FEM simulation of steam turbine blades materials hot forming, ADVANCES IN MATERIALS AND PROCESSING TECHNOLOGIES XV Book Series: Materials Science Forum Volume: 773-774 Pages: 79-88 Published: 2014, DOI: 10.4028/www.scientific.net/MSF.773-774.79

[3]-Džugan, J., Konopik, P., Rund, M., Prochazka, R., Determination of local tensile and fatigue properties with the use of sub-sized specimens, (2015) American Society of Mechanical Engineers, Pressure Vessels and Piping Division (Publication) PVP, 1A.

[4]-Džugan, J., Procházka, R., Konopík, P., Micro-tensile test technique development and application to mechanical property determination, (2015) ASTM Special Technical Publication, STP 1576, pp. 12-30.

[5]-Maresova M., KONOPÍK P.: Tensile Testing at High Strain Rates Using DIC Method, conference proceedings METAL 2013, 15-17. May, Brno, Czech Rep. ISBN 978-80-87294-39-0.

[6]-Konopik, P., Dzugan, J., Rund, M.: Dynamic Tensile And Micro-Tensile Testing Using Dic Method, Metal 2014, May 21st - 23rd 2014, Brno, Czech Republic, ISBN 978-80-87294-52-9.

[7]-Konopík P., Džugan J.: Determination of Tensile Properties of Low Carbon Steel and Alloyed Steel 34CrNiMo6 by Small Punch Test and Micro-Tensile Test, 2st International Conference SSTT, 2.10.-4.10.2012, Ostrava, Czech Republic, ISBN 978-80-260-0079-2

[8]-Keating T.J., Wolf P.R., Scarpace F.L. An Improved Method of Digital Image Correlation. *Photogrammetric Engineering and Remote Sensing* 1975, y. 41, No. 8, p. 993-1002.

[9]-Kumar K, Madhusoodanan K., Rupani B. B.,: Miniature Specimen Technique as an NDT Tool for Estimation of Service Life of Operating Presurre Equipment, International Conference & Exhibition on Pressure Vessel and Piping, February 7-9, 2006, Chennai, India.

[10]-Opbroek E. at al:, Recommendations for dynamic Tensile Testing of Sheets Steels. International Iron and Steel Institute, 2005. [online] http://c315221.r21.cf1.rackcdn.com/HighStrainRate_Recommended_Procedure.pdf

[11]-Rund, M., Procházka, R., Konopík, P., Džugan, J., Folgar, H., Investigation of Sample-size Influence on Tensile Test Results at Different Strain Rate, (2015) Procedia Engineering, 114, pp. 410-415.

[12]-Sutton M.A., Orteu J.-J., Schreier H. W. Image Correlation for Shape, Motion and Deformation Measurements. New York: Springer Science+Bussines Media, 2009.