

**METHODOLOGY FOR INVESTIGATION OF CREEP PROPERTIES USING SUB-SIZE SPECIMEN**<sup>1</sup>Eva CHVOSTOVÁ, <sup>1</sup>Pavel KONOPÍK<sup>1</sup>COMTES FHT a.s., Průmyslová 955, 334 41 Dobřany, Czech Republic, EU  
[echvostova@comtesfht.cz](mailto:echvostova@comtesfht.cz), [pkonopik@comtesfht.cz](mailto:pkonopik@comtesfht.cz)<https://doi.org/10.37904/metal.2023.4701>**Abstract**

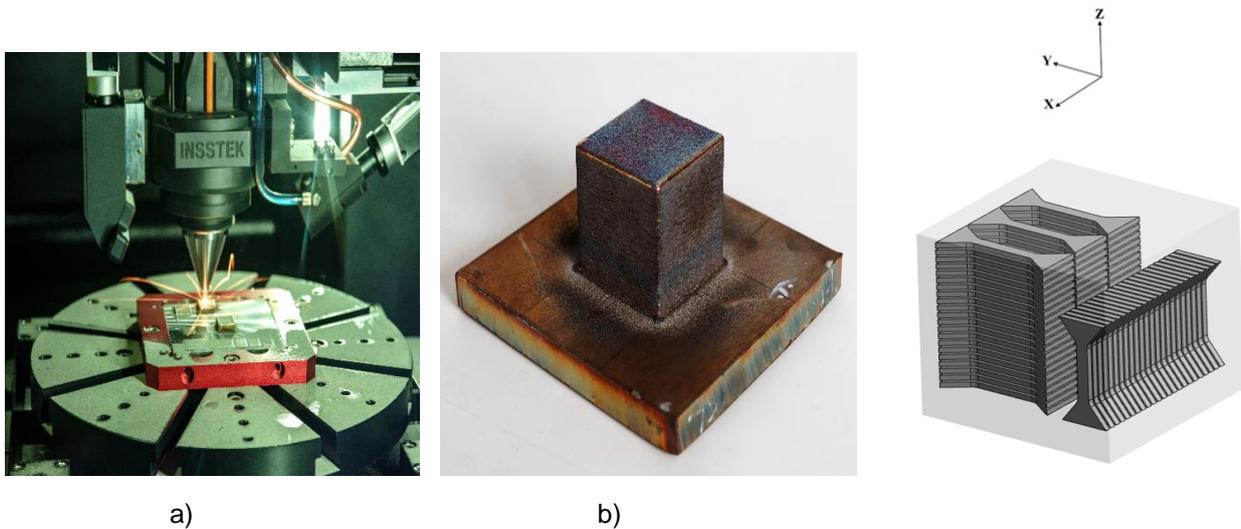
One of the methods of evaluating the mechanical properties of a material in the case of its limited amount is the use of techniques that employ the miniaturized test specimens. The basic properties used mostly for residual life evaluation are tensile strength, impact notch toughness or impact notch toughness transition curve, fracture toughness, creep and high cycle fatigue. This paper investigates the creep properties using sub-size specimens with a cross section 2x1mm. The methodology of the creep properties evaluation is presented. Materials manufactured using laser-directed energy deposition process Inconel 718, and 10Cr-3W-3Co-1Mo creep-resistant steel without nitrogen content were tested at a temperature of 650 °C.

**Keywords:** Creep test, sub-sized samples, IN718**1. INTRODUCTION**

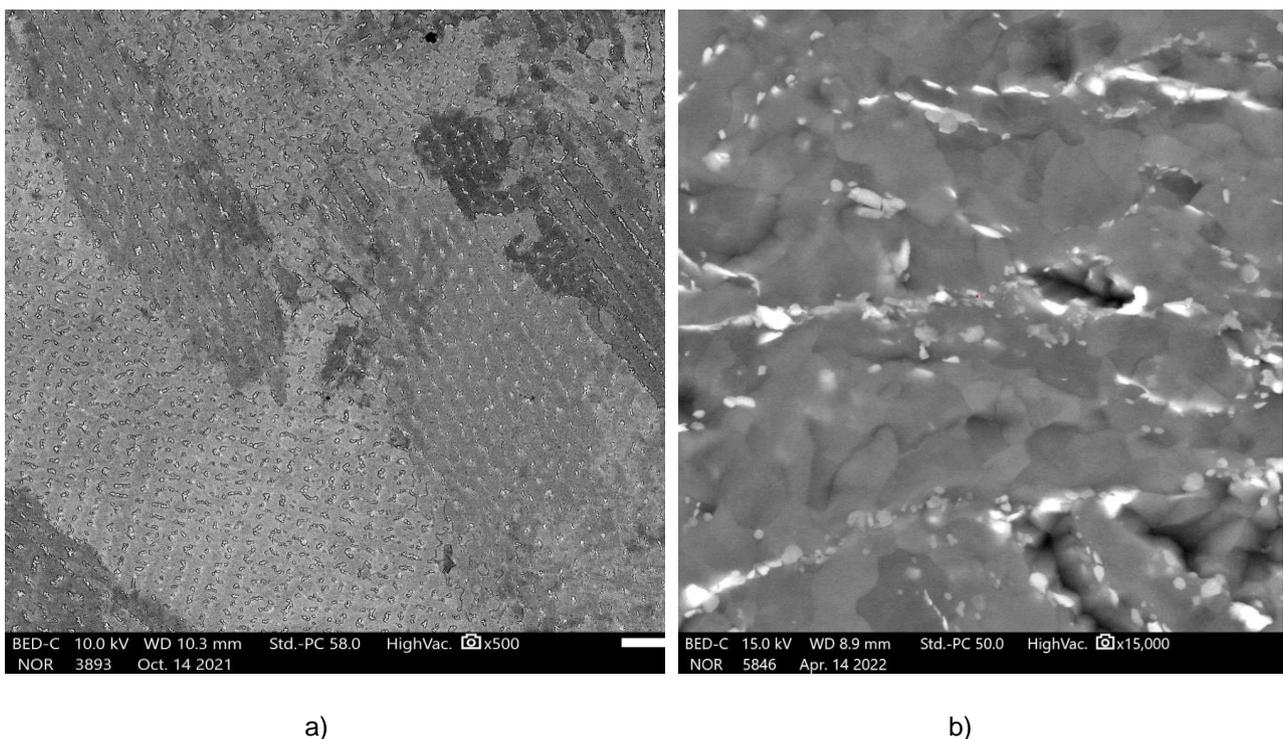
Mechanical properties of materials can be evaluated using miniaturized test specimens in the cases when limited amount of experimental material is available. Tensile strength, impact notch toughness, fracture toughness, creep, and high cycle fatigue are the most basic and important properties for lifetime assessment of the component. Each of these techniques is differently sensitive to the degradation process of the material. Therefore, it is necessary to carefully choose the most appropriate method considering amount of available material and various specimen size for each test. This study focuses on the creep properties investigation of additively manufactured nickel-based alloy Inconel 718 using laser-directed energy deposition (L-DED) method and 10Cr-3W-3Co-1Mo (nitrogen free creep resistant steels). Miniaturized specimens dedicated to creep testing, with a cross-section of 2x1mm were subjected to the uniaxial tensile loading at elevated temperature of 650 °C. The methodology of creep test performance is discussed, and emphasis is placed on practical test result applications.

**2. EXPERIMENTAL MATERIAL**

Nickel based alloy IN718 [1] and nitrogen free creep resistant steel 10Cr-3W-3Co-1Mo were selected for the experiment. These materials are commonly used in power plants thanks to their high temperature resistance. In the case of IN718, the specimens were extracted from the experimental block (35x35x50 mm) manufactured using (L-DED) process. The test specimens were extracted in YXZ (horizontal) and ZYX (vertical) orientation according to the cutting plan depicted in **Figure 1**. The description of the structure is shown in **Figure 2**. Inconel 718 observed in as-deposited state. The material was printed by DED, the building direction is oriented upwards in the image. The microstructure is composed of coarse grains distinguishable by different shading in the image as well as different patterns of the intermediate phases. These are visible as bright particles of irregular shape evenly distributed in the matrix. Intermetallics were formed in the interdendritic regions from the last bits of solidifying metal and therefore shows the orientation and spacing of the dendrites [2,3]. 10Cr-3W-3Co-1Mo creep resistant steel exposed at 650 °C for 1000 hours. Sub grains are visible in the matrix. Bright particles of intermediate phases and carbides outlines boundaries of original martensite crystals.



**Figure 1** Laser directed energy deposition process (a), experimental block (b), cutting plan (c)



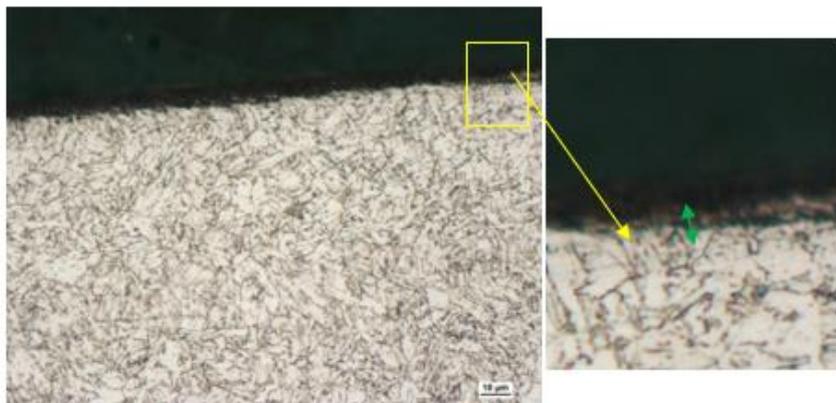
**Figure 2** Microstructure of the 3D-printed IN718 (a), Microstructure of the material 10Cr-3W-3Co-1Mo (b)

### 3. MATERIAL SAMPLING AND SUB-SIZE SPECIMENS

In order to produce the specimens from experimental steel 10Cr-3W-3Co-1Mo a special procedure was applied to the service component simulating a real process of the material extraction. A portable Electric Discharge Sampling Equipment (EDSE) [4] 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, see **Figure 3**. The influence of electro-erosive machining on the work piece is demonstrated in the **Figure 4**. The depth of the affected layer appeared to be slightly less than 10  $\mu\text{m}$  thick.

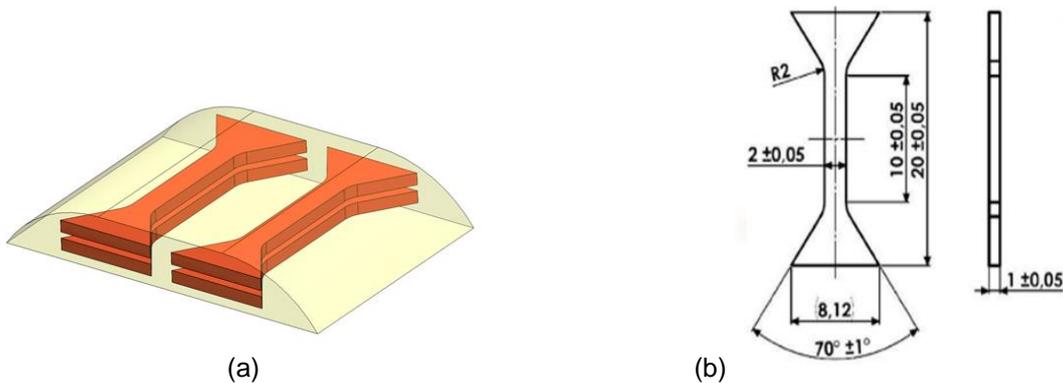


**Figure 3** The set-up of EDSE; whole device set up on the left and right is specimen detail



**Figure 4** Influence of EDS machining on microstructure – affected depth is less than 10  $\mu\text{m}$

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 production. Specimens outline and cutting scheme within the extracted piece of material can be seen in **Figure 5 a)**. For 10Cr-3W-3Co-1Mo material, test specimens with a diameter of two millimeters [5] were used. Both dimensions are based on the shape of the sample with dimensions of 20x20x4 mm, that can be extracted from real in service components by semi-destructive device EDSE (the Electric Discharge Sampling Equipment). All sub-sized specimens were subsequently machined from the extracted material. Creep tests were carried out on these test specimens whose geometry is depicted in **Figure 5 b)**.



**Figure 5** Specimens cutting scheme within EDSE extracted material (a) and geometry samples used for creep testing (b)

## 4. CREEP TESTS

### 4.1 Performing creep tests

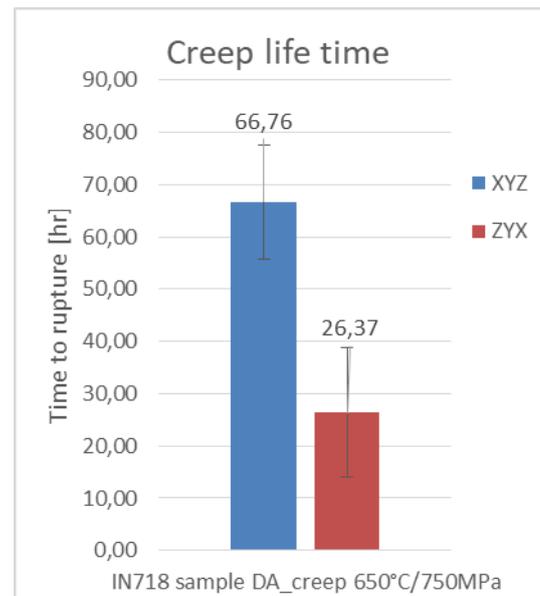
Creep tests test bars were executed with the use of modified creep machines equipped with furnace, strain and temperature measurement [6]. Test specimens of material IN718 designation DA were tested with five replicates per orientation at creep conditions of 650 °C test temperature and the constant applied load of 750 MPa. Further tests were carried out at the same temperature of 650 °C, but at different constant applied loads.

### 4.2 Results of creep tests

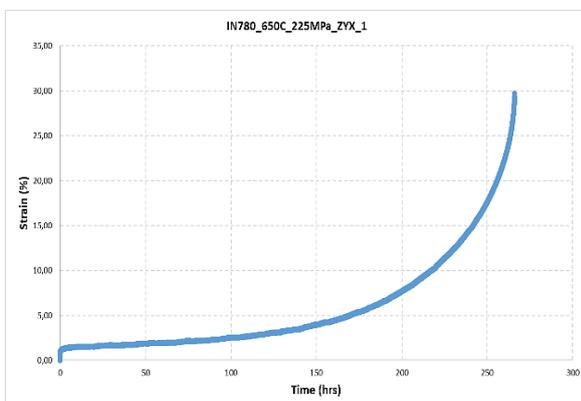
Measured creep lifetime in horizontal and vertical orientation of deposited IN718 is depicted in **Figure 6**. The column graph shows the average creep life time value and standard deviation based on the five measurements. It is evident that deposited IN718 is more durable to the creep load in horizontal orientation with the average of  $66.8 \pm 10.91$  hr. Vertically oriented specimens showed more than twice lower creep life with the average value of  $26.4 \pm 12.33$  hr.

For all tested specimens a creep curves (time-strain) were recorded, see the representative example in **Figure 7 a)**. Based on these records a creep rate is possible to evaluate for each specimens, which is demonstrated in **Figure 7 b)**.

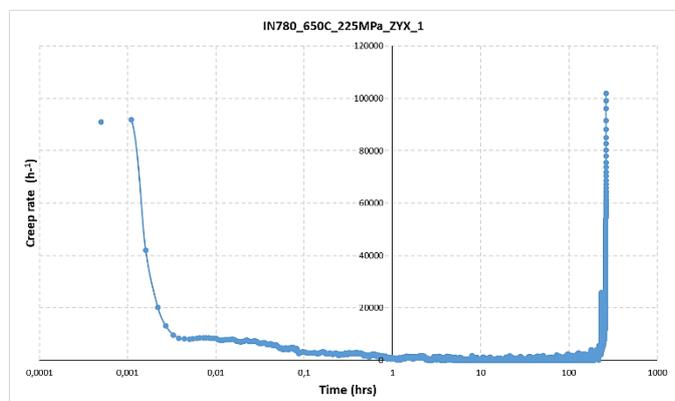
Creep test results of creep resistant steel 10Cr-3W-3Co-1Mo are depicted in **Figure 8** using Larson-Miller creep diagram. Together with the data based on the sub-sized specimen measurement (blue points) standard specimens results (orange points) are presented as a reference. In addition, results of material P92 presented by the EUROPEAN CREEP COLLABORATIVE COMMITTEE are plotted. The comparison of the plotted curves demonstrated that the results of sub-sized specimens are shifted above the standardized, thus for the certain stress value the miniaturized specimens give larger LM parameter, which corresponds to the longer estimated lifetime.



**Figure 6** Comparison of creep test results in XYZ and ZYX direction of IN718 DA sample

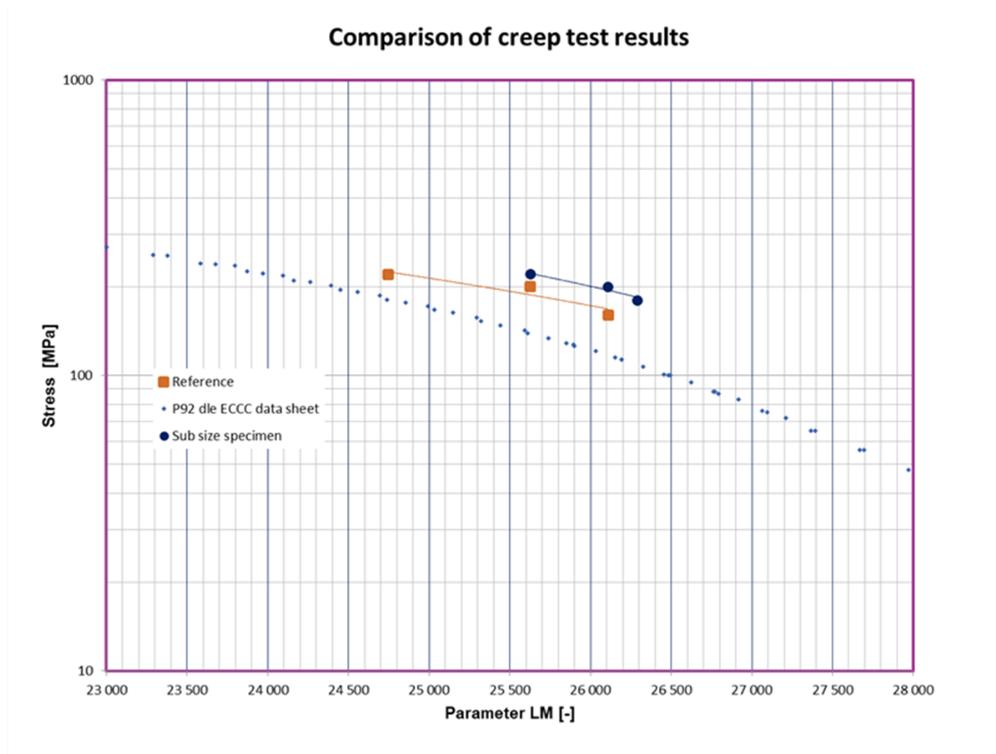


a)



b)

**Figure 7** Sample IN780, 650 °C/225MPa creep curve (a), Creep rate (b)



**Figure 8** Comparison of creep results for material 10Cr-3W-3Co-1Mo

## 5. CONCLUSION

The paper deals with possibilities of creep properties determination with the use of miniaturized specimens. Standard creep specimen results are compared with results obtained from mini-creep specimens. The standardized specimens provide reliable results, however it is usually impossible to machine them out of real in-service components. Therefore, miniaturized creep specimens were proposed since they keep the same loading mode as standard creep specimens, while material demand for their production can be covered by the amount of material that can be extracted from service component. The dimensions of creep mini-samples proposed here are based on material volume that can be extracted by available EDSE sampling device. The same specimen's geometry can also be used for the evaluation of anisotropy in samples produced by additive technology. The results showed in detail the difference in creep properties in the two investigated directions.

Despite the fact that the trend of plotted data in Larson-Miller diagram for tested creep resistant steel was the same, the data for miniaturized specimens were clearly shifted above the standard one, which affects the evaluated lifetime of the material. However, there is a need to test additional specimens in order to gain more accurate results since only 3 miniaturized specimens were tested.

It was demonstrated that is possible to use sub-size samples to determine the creep properties.

The anisotropy of samples produced by additive technology (3D printing) can be well described with the help by sub-size test samples.

Sub-size test samples can also be produced from extracted samples using semi-destructive sampling (EDSE) and thereby e.g. determine the residual service life of components using current creep mechanical properties

## ACKNOWLEDGEMENTS

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