A Review of miniature specimen tensile test method of tungsten at elevated temperature

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Abstract
Tungsten is the key material for high heat flux components of divertor in nuclear fusion reactor. The temperature range of high heat flux components is around 1200-2200 °C. Very limited database is available for tungsten at higher temperature. So, characterization of tungsten material at higher temperature is very much essential. Miniature specimen tensile testing technique is useful to characterize tungsten for life assessment of plasma facing component, characterization of neutron irradiated specimen and characterization of various brazed and diffusion bonded joint, characterization of sintered pallets etc. The purpose of the review of small size specimen test techniques is to carry out design of miniature specimen for high temperature tensile testing of tungsten, effect of geometry on material properties and validation of test results using finite element analysis.

Index Terms **- Miniature specimen, tungsten material, hot tensile test, finite element analysis, Abaqus.**

I. Introduction

Tungsten is the key material as a plasma facing material (PFM) for plasma facing components (PFCs) such as divertor targets and dome[1].In fusion reactors, plasma facing materials are subjected to high heat loads radiating from the plasma. In particular, divertor components are subjected to steady state heat loads of ~10 MW/m^2[2]. Tungsten has been selected as armor for the divertor upper vertical target, dome, cassette liner, and for lower base because of its unique resistance to ion and charge-exchange particle erosion in comparison with other materials [3].There is a very limited database for the tungsten grades within the temperature range of interest to ITER [3]. G. E. Lucas has been studied different miniature specimen test techniques like instrumented micro hardness, bulge, shear punch, indentation creep and miniaturized fractured test for obtaining strength, ductility, time-dependent flow, fracture behavior of material [4]. D. Finarelli has been carried out miniature specimen test using small punch test to determine mechanical properties ranging from elastic behavior [5]. J. Reiser has been worked on miniature specimen test by charpy impact test to characterize tungsten material in as-received condition and recrystallized condition [6]. T. Yamamoto has been carried out cyclic ball indentation method for different Fe-Mn-Cu-C alloys to study the behavior of material with and without annealing treatment [7].Recent progress towards small specimen test techniques are fatigue test, fracture toughness test and crack growth rate test of RAFM steel in fusion reactor,2015 [8].

II. Review on small specimen test techniques

1. Effect of specimen geometry on tensile properties

R. L. Clueh [9] has been worked on the different types of miniature tensile test specimens for fusion reactor irradiation studies. Three miniature sheet-type tensile specimens and a miniature rod tensile specimen were being used in irradiation studies for fusion reactor materials. Consequently, a comparison of tensile properties determined with these different specimens was made by testing cold-worked and solution-annealed Type 316 stainless steel sheet and rod at RT, 300 °C and 600 °C.

In this paper, three miniature sheet type specimens SS-1, SS-2 and SS-3 are designed. SS-1 specimen was developed in fast breeder reactor program for irradiation in experimental breeder reactor as shown in figure 1.

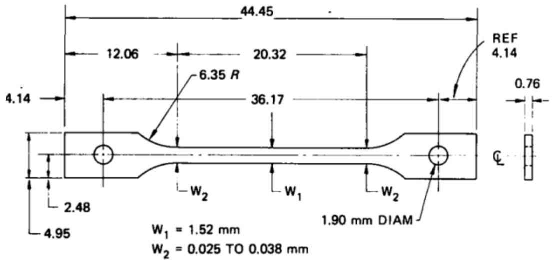
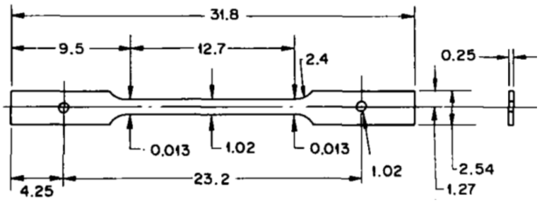
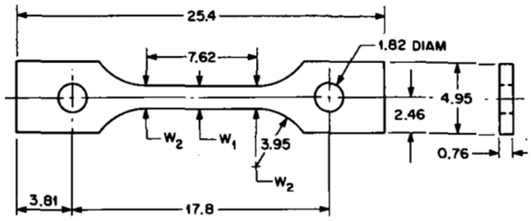


Figure 1- (a) Miniature SS-1 sheet tensile specimen



(b) Miniature SS-2 sheet tensile specimen



(c) Miniature SS-3 sheet tensile specimen

The tensile properties determined with the different specimens were in good agreement when differences in grain size, cold work and gage length taken into account. Variation in YS and UTS for a given specimen, as measured by standard deviation, was usually well below 10%. The largest standard deviations were observed for SS-2 specimen. Uniform elongation of SS-3 specimen is greater than all other specimens but tensile strengths obtained from all specimens are almost equal so any of the specimen types is adequate.

2. Effect of specimen thickness on tensile properties

Byun T. S. et al. [10] have been studied on the effect of specimen thickness on tensile properties of SA508 Cl.3 steel with the thickness range of 0.2-2 mm of miniature specimens. This study has been carried out to determine the minimum thickness requirement and a correlation with the properties of a standard specimen for practical application. The tensile properties are independent of specimen thickness when the thickness is larger than the critical thickness. The chemical composition of steel is given in table 2.1. The steel-making process of this forging was aluminum-added vacuum silicon deoxidation method and the quality heat treatment after the ring forging included austenitizing-water quenching and tempering at 660 °C for 31 hours. Microstructure of steel was mainly tempered bainite, and the prior austenite grain size was about 40 µm.

Table 1 Chemical composition (wt. %) of the SA508 Cl.3 RPV Steel

C	Mn	Si	Ni	Cr	Mo
0.21	1.36	0.24	0.92	0.21	0.49
V	Al	Cu	P	S	
0.005	0.022	0.03	0.007	0.002	

The miniature specimens with various thicknesses were prepared by electric discharge machining (EDM) and multistep polishing. The thicknesses of the specimens were varied from 0.1 mm to 2.0 mm. The gage length and the width of the specimen were 10 mm and 3 mm respectively. Tension test were carried out in an Instron static testing machine at a crosshead speed of 1mm/min, which gives a strain rate of 0.00167 sec^{-1} in uniform deformation region at room temperature.
Results show the thickness effect on the yield and ultimate tensile strength in the miniature specimens. Both the yield strength and ultimate tensile strength are nearly constant in the thickness range of 0.2–2 mm, and the value of thick specimens is almost the same with the standard round specimens. Therefore the critical thickness for the test material is regarded as 0.2 mm. The critical thickness of miniature tensile specimen has been expressed by the ratio of thickness to grain size. The ratio of critical thickness to grain size of type 304 and 316 austenitic stainless steels is 4 and 6 respectively. The ration is about 5 for the coarse grained copper and aluminum. The grain size of the test material was about 40µm, means that the critical thickness of the test material is also about 5 times as large as the grain size.

3. Effect of L/W ratio on tensile properties

O.N. Pierronet al. [11] have been worked on tensile specimen geometry and constitutive behavior of Zircaloy-4 alloy. The influence of tensile specimen geometry on the deformation behavior of flat Zircaloy-4 tensile specimens has been examined for gauge length-to-width ratios that range from 1:1 to 4:1. Tests were conducted at room temperature and an initial strain rate of 10^{-3} s^{-1}. The rolling direction of the sheet was taken as the extrusion direction of tube materials. The tensile tests performed on sheet material with the tensile axis oriented transverse to the rolling direction.

Table 2 A comparisons of the different tensile specimen geometries

Specimen	***Gage length l (mm)***	***Gage width w (mm)***	***Fillet radius R (mm)***
1:1	10	10	7.5
3:2A	15	10	5
3:2B	15	10	7.5
4:1	40	10	10

Specimen geometries with dimensions of different specimen are compared in table. Specimen geometry has only minor effects on the values of the yield stress, tensile strength, apparent uniform strain at maximum load, and strain-hardening exponent. However, in all geometries but the 4:1 configuration, diffuse necking occurs before maximum load. As a result, strain distributions at maximum load are uniform only in the 4:1 geometry. The elongation to failure is also affected by specimen geometry with the shorter gauge sections exhibiting much higher total elongation values as shown in figure 2.

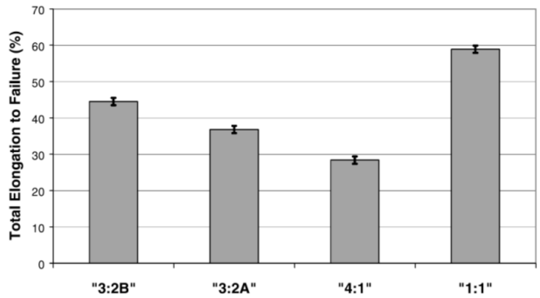


Figure 2-Total elongations to failure as a function of specimen geometry

the differences between the 4:1 geometry and the shorter 3:2 geometries are small with only the total elongation being strongly affected by the geometry; in this case, the shorter gauge-section specimens exhibits much greater total elongation values. 4:1 gage length to width is best suitable for tensile strength and elongation result.

4. Effect of specimen size on tensile properties

Kundan Kumaret al., [12] 2014 have been estimated the use of miniature tensile specimen for measurement of mechanical properties. They evaluated the mechanical properties of miniature and sub-size tensile test specimens, which can be useful for life estimation of any in-service-equipment and for development of new materials. Both these applications intend to use very small amount of material for evaluation of the mechanical properties. Apart from various types of novel techniques developed worldwide, the evaluation of mechanical properties from a miniature tensile test has a greater advantage as it is a direct method of measurement of mechanical properties.

This paper is limited to the evaluation of mechanical properties using the miniature tensile test specimen. In order to ensure the usefulness of miniature test techniques it is important to have comparable results of miniature tests and conventional tests with respect to the mechanical properties, viz. UTS, YS and elongation. Two different types of tensile test specimens; namely type-I (conventional round) specimen, type-II (sub size flat) specimen have been tested and compared with the results of type-III (miniature specimen) as shown in figure 3.86 mm overall length with 30 mm gage length having 6 mm diameter of standard round specimen, 47 mm overall length with 9.5 mm gage length having 3 mm wide and 1 mm thick of sub size specimen and the miniature specimen having 3 mm gage length, 1 mm wide and 0.3 mm thick of overall length of 11.3 mm were designed.

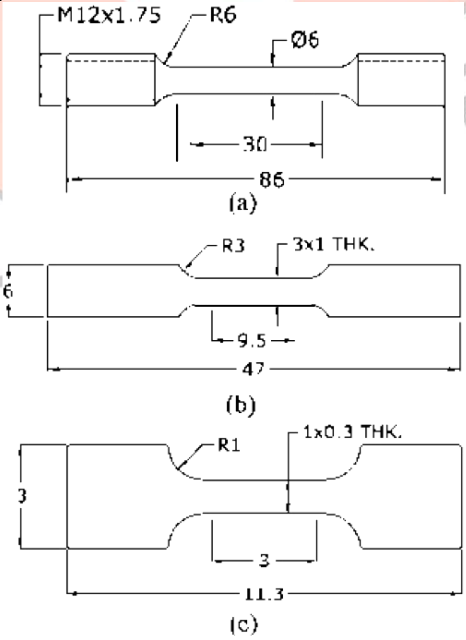


Figure 3-Dimensional details of tensile test specimens

Three alloy materials 20MnNiMo55, CrMoV and SS304 LN are used for miniature tensile test. As a result of comparison of miniature specimen with standard specimen indicated that UTS value of miniature specimen is 2-4.5 % less than standard specimen and YS value is 0.2-6 % for same.

Figure 4- Temperature dependence of fracture strength and yield strength

5. Effect of temperature on tensile properties

Kenta Sasaki et al., [2] 2014 have been carried out effect of temperature on tensile properties of pure tungsten and K-doped tungsten at temperatures from 25-700°C for plasma facing component in nuclear fusion reactor. Divertor components are exposed to steady state heat load of ~10 MW/m^2, but also to thermal shocks of very short duration. K doped W and pure W were fabricated by powder sintering and hot rolling with rolling reduction ratio of 80% and heat treatment at 900°C for 20 min for stress relief. Tensile test were carried out in vacuum at a pressure of 10^{-3} Pa in temperature range of RT to 700°C, at strain rate from 10^{-5} to 10^{-1} s^{-1}. The temperature dependence of yield strength and fracture strength obtained are shown in figure 5. It has been concluded that yield strength of both material increased with decreasing temperature and fracture strength of both material increased with decreasing temperature.

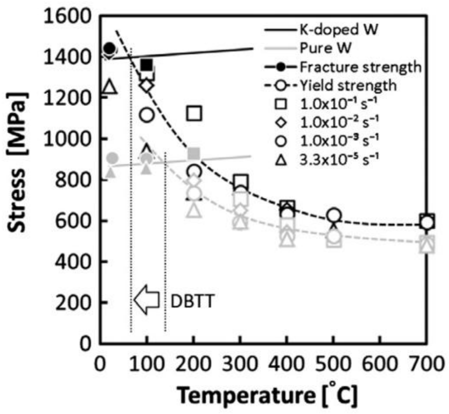


Figure 5- Temperature dependence of UTS of Pure W and K-doped W

Figure 6 shows the strain rate sensitivity against temperature. The strain rate sensitivity of both materials was higher at lower temperature and decreased with increasing temperature. Strain rate sensitivity is used to determine the quantitative measurement of yield strength with respect to strain rate.

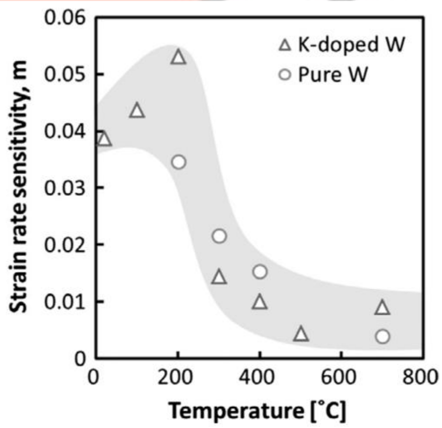


Figure 6-Temperature dependence of strain rate sensitivity

6. Effect of strain rate on tensile properties

Kenta Sasaki et al. 2014 [2] have been carried out effect of strain rate on tensile properties of pure tungsten and K-doped tungsten at strain rate range of 10^{-5} to 10^{-1} s^{-1} for plasma facing component in nuclear fusion reactor. Figure shows the yield strength against strain rate for K doped and pure tungsten. The yield strength of both materials increased with increasing strain rate. The strain rate dependence of the fracture strain determined from the stress–strain curves is shown in figure for K-doped W and pure W. The fracture strain of K-doped W showed a sharp decrease at 100 °C, indicating that the DBTT increased with increasing strain rate. For pure W, a similar sharp decrease in fracture strain was observed at 200 °C. Therefore, K-doped W showed ductility at lower temperature and a lower DBTTcompared to pure W. On the other hand, at temperatures higher than 300 °C, the fracture strain of K-doped W and pure W were similar, and both increased with increasing strain rate.

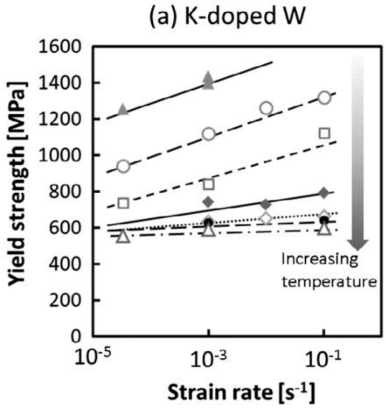


Figure 7-Strain rate dependence of yield strength (K-doped tungsten)

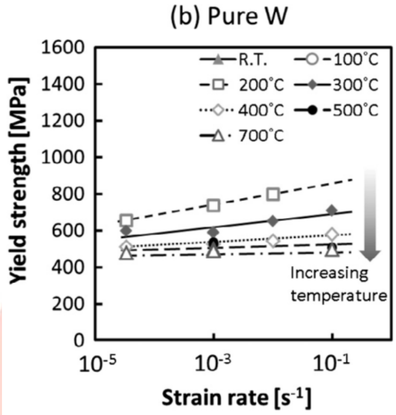


Figure 8-Strain rate dependence of yield strength (pure tungsten)

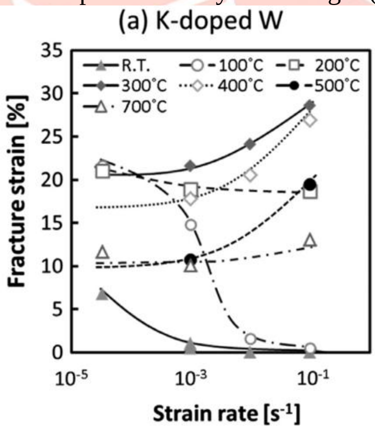


Figure 9-Strain rate dependence of fracture strength (K-doped tungsten)

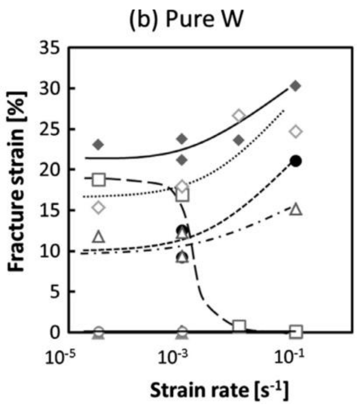


Figure 10-Strain rate dependence of fracture strength (pure tungsten)

Mokoto Fakuda et al., [1] 2015 have been carried out the effect of strain rate on tensile properties such as yield stress and fracture strain of W-3%Re and K-doped W-3%Re plates in the temperature range from room temperature (RT) to 300 °C. . K doped W and pure W were fabricated by powder sintering and hot rolling with rolling reduction ratio of 80% and heat treatment at 900°C for 20 min for stress relief. The dimensions of the gauge section of the specimens were 5mm long, 1.2 mm wide and 0.5 mm thick. Specimens were cut out in the rolling direction of the plate by electro discharge machining.

Surface was mechanically polished to #1500. Tensile tests were performed in vacuum and in a temperature range from RT to 300 °C using an electromotive testing machine. The strain rate was in the range from 10^{-3} to 10^{-1} s^{-1}. The strain rate dependence of the yield stress in K-doped W-3%Re and W-3%Re are shown in figure. The yield stress in both K-doped W-3%Re and K-doped W tended to increase with increasing strain rate, and this trend was more clearly observed in W-3%Re than in K-doped W- 3%Re. K-doped W-3%Re showed lower strain rate dependence as compared to W-3%Re at 200 and 300 ◦C.

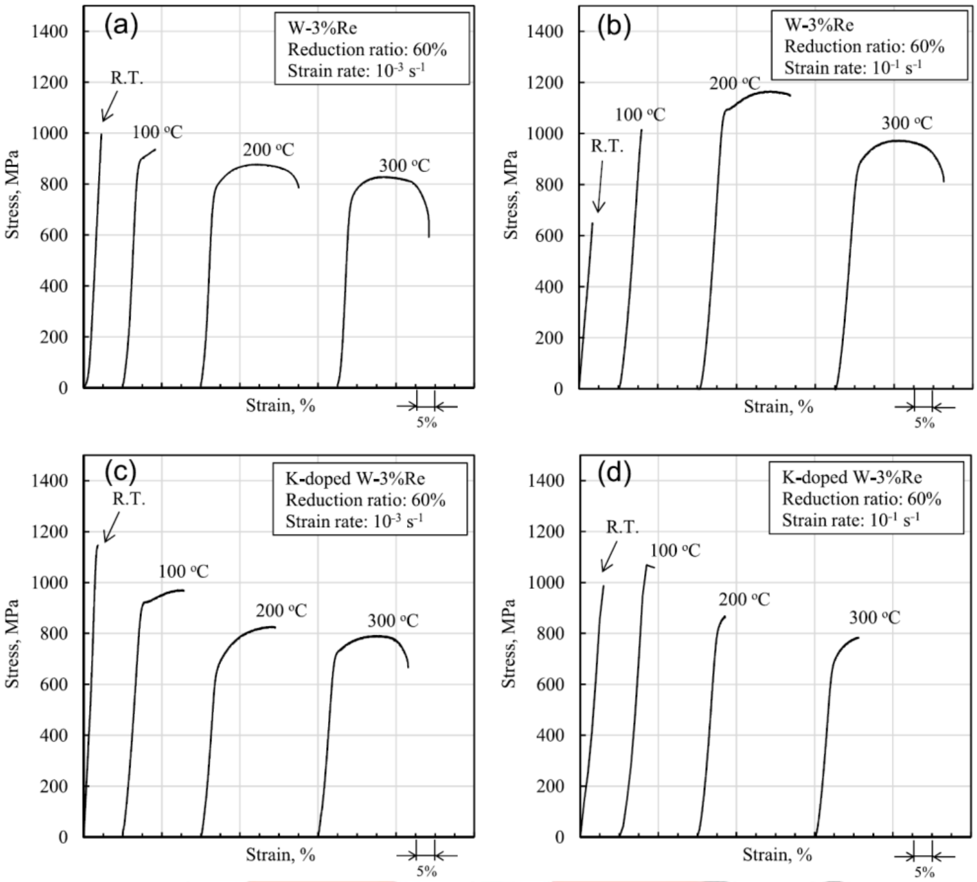


Figure 11- Engineering stress–strain curves obtained by tensile test of W-3%Re and K-doped W-3%Re.

III. Review on finite element analysis of miniature specimen

Validation of experimental results of tensile testing of miniature specimen is most important. Finite element analysis procedure is one the most suitable method for validation test results.

1. Inverse finite element analysis

Asif Husainet al., [16] 2004 have been worked on an inverse finite element procedure for the determination of constitutive tensile behavior of materials using miniature specimen. An experimental and a computational study of small punch test using circular disk shaped miniature specimen, through inverse finite element procedure. An inverse finite element procedure is developed and clubbed with ABAQUS computer code for the determination of constitutive tensile behavior of materials. The proposed inverse technique is based on the small punch experimental load vs. displacement curve. Small punch tests (SPT) are conducted on circular disk shaped specimen (10 mm diameter, 0.5 mm thick) made from three different steels.

Three materials (a) Chromium hot work steel (H11), (b) medium carbon steel (MS), (c) non-shrinkage die steel (D3) are used to finite element and experimental methods. Figure shows the typical load-deformation curves obtained from inverse finite element method.

G. Partheepanet al., [17] 2008 have been worked on finite element application to estimate in-service material properties using miniature specimen. The studies have been conducted in a chromium (H11) steel and an aluminum alloy (AR66). The output from the miniature test *viz.* load-elongation diagram is obtained and the finite element simulation of the test is carried out using a 2D plane stress analysis. The results are compared with the experimental results. It is observed that the results from the finite element simulation corroborate well with the miniature test results.

The finite element modeling computations were conducted using the finite element code, ABAQUS. The test specimen is modeled with eight noded quadratic quadrilateral plane stress elements because the thickness of the body or domain is small relative to its lateral (in-plane) dimensions. The stresses are functions of planar coordinates alone, and the out-of-plane normal and shear stresses are equal to zero. The free meshing option is used for the meshing of the test specimen. The sufficiency of the mesh fineness was confirmed by testing the finer one which is made of 6000 elements.

The loading pins were treated as 2-D rigid bodies with a low friction coefficient of 0.01. In the elastic analysis of simulation, the material properties of the columns were defined by density, elastic modulus and Poisson's ratio. In the nonlinear analysis stage, material nonlinearity or plasticity was included in the FEM using a mathematical model known as the incremental plasticity model. In the finite element simulation of miniature dumb-bell specimen, the plastic properties are defined together with the isotropic hardening rule. It means that the yield surface size changes uniformly in all directions such that the yield stress increases in all stress directions as plastic straining occurs.

2. Comparison with experimental load-deformation curves

Sunil Goyal[15] 2010 have been worked on Finite element analysis of shear punch testing and experimental validation at Indira Gandhi Centre for Atomic Research, Kalpakkam. In this work, finite element analysis (FEA) of the shear punch testing is carried out to study the specimen deformation up to yielding and the results are compared and validated with experimental data for four different materials

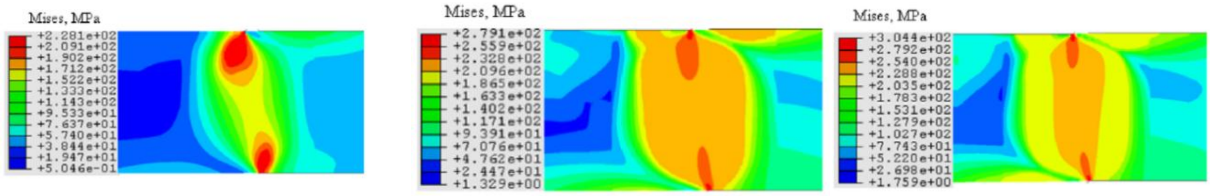


Figure 12-Von Mises stress contour profiles before yield, at yield and after yield

The elastic portion of the FEA generated load–displacement curve overlaps with the corresponding experimental curve only when the fixture compliances are eliminated in experiments as shown in figure. Based on through thickness plasticity in the FEA study, the shear yield stress estimated at an offset of 0.15% of normalized displacement compares well with the experimentally determined shear yield strength and satisfies the von mises yield relation $\sigma_{ys} = 1.73\ \tau_{ys}$.

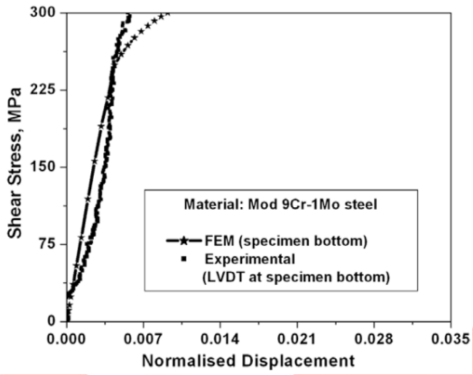


Figure 13-Comparison of FEM results with experimental load –deformation curve

Kundan Kumaret al., [12] 2014 have been worked on use of miniature tensile specimen for measurement of mechanical properties using finite element analysis. The finite element modeling computations were conducted using a commercial finite element modeling software. The test specimen is modeled with four noded quadrilateral plane stress elements because the thickness of the body or domain is small relative to its lateral (in-plane) dimensions. Geometric nonlinearity was also modeled on top of material non linearity. In order to represent the experimental setup, the loading pin was simulated for providing load to the specimen. The loading pin was treated as 2-D rigid body with friction coefficient of zero.

In the elastic analysis of simulation, the material properties of the columns were defined by elastic modulus and Poisson's ratio. In the nonlinear analysis stage, material nonlinearity or plasticity was included in the FEM using a mathematical model known as the associated plasticity flow rule with von-Mises yield criterion. In the finite element simulation of miniature specimen, the plastic properties are defined together with the isotropic hardening rule. It means that the yield surface size changes uniformly in all directions such that the yield stress increases in all stress directions as plastic straining occurs. In the incremental plasticity model true stresses (t) and true plastic strains (tp) of a conventional test specimen were specified. Figure 14 shows the comparison of experimental and finite element analysis results

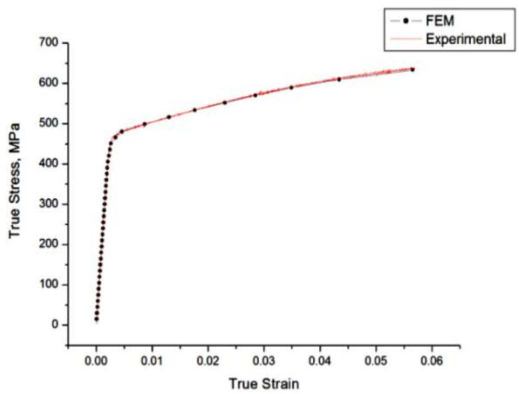


Figure 14- Comparison for true stress- true stain curves

IV. Conclusion

Literature review reveals that the researchers have carried out most of the work on miniature specimen test on different steels, aluminum alloys and other metallic alloys, but negligible work done on miniature specimen test for pure tungsten material. There is a very limited database for pure tungsten material available within the operating range up to 2000 °C for the application of plasma facing component in divertor. So, tungsten material must be characterized at higher temperature. Miniature specimen test technique is the best suitable method for characterization of in-service component as availability of material is limited in size.

V. Acknowledgement

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