Development of Ultra Sub-size Tensile Specimen for Evaluation of Tensile Properties of Irradiated Materials

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Abstract. The idea of using small specimens for mechanical testing had actually originated in the nuclear industry to cater to the irradiation material testing and reactor surveillance programs. Small or miniaturized specimens ensure efficient use of available irradiation volume in nuclear reactors, reduce uncertainty in irradiation parameters due to flux and temperature variations and also reduce radiological hazard during testing. A procedure for tensile testing employing a miniature tensile specimen called ultra sub-size (USS) tensile specimen carved out of a 10.0 mm diameter and 0.5mm thick disc sample has been developed and standardized. The geometry of the specimen was optimized using Finite Element Analysis (FEA). USS specimens along with ASTM standard and sub-size specimens were tested on a range of fast reactor structural materials for comparison of mechanical properties. Due to difficulty of employing extensometer on a small gage length of 3.0mm, digital image correlation (DIC) was employed for strain measurement which has added advantage of permitting determination of strain distribution across the gage length. The strain obtained through DIC was co-related with that obtained from the cross-head displacement of the UTM. The results obtained from tensile testing of ultra sub-size specimen at ambient and elevated temperatures were found to be consistent and comparable with those obtained from standard specimens for a wide range of alloys examined. The results of this study show that USS tensile specimen geometry can be reliably employed for mechanical property evaluation of irradiated structural materials.

Key Words: Miniature tensile testing, Digital Image Correlation (DIC), Finite Element Analysis (FEA).

1. Introduction

Development of economically viable nuclear reactors relies heavily on development of new materials which can sustain reactor conditions for prolonged periods. Irradiation damage is primarily evaluated by tensile testing of new alloys irradiated in reactor operation conditions. Tensile testing of specimens extracted from core components of a nuclear reactor also exhibits the extent of irradiation damage. It helps in condition monitoring of components as well as life extension of permanent structural components. Tensile testing of irradiated material either through irradiation of specimens in a reactor or extraction of specimens from reactor components gives mechanical properties like ultimate tensile strength, yield strength and elongation.

Tensile testing of irradiated material poses challenges such as radiological hazard on account of activation. The space available in the reactor for irradiation is limited. Thus employing conventional tensile specimens having overall size in few centimetres is not an effective way of extracting mechanical properties of irradiated material. The neutron and temperature fields inside a reactor are varying, more so in fast reactor owing to compact core. This results in non-uniform damage across gage length and introduces uncertainty in the mechanical properties evaluated of such specimen. Small or miniaturized specimens ensure efficient use of available irradiation volume in nuclear reactors; reduce uncertainty in irradiation parameters due to flux and temperature variations and also reduce radiological hazard during testing [1].

Conventional tensile specimens recommended by international standards like ASTM, DIN, ISO etc have gage length and width in the range of few centimetres and thickness of few millimetres. As miniaturization of specimens is beneficial for testing of irradiated material, other investigators have utilized miniaturized specimens having gage lengths varying from 3 to 20mm, widths varying from 0.2 to 3mm and thicknesses varying from 0.2 to 1mm. Currently a sub-size specimen of 12.5mm gage length, 3mm width and 1mm thickness is employed for mechanical property evaluation of irradiated material. This paper deals with development of an ultra sub-size (USS) tensile specimen carved out of 10mm diameter and 0.5mm thickness disc having 1.5mm gage width and 3mm gage length. Constraint for further miniaturization is the ratio of thickness to avg. grain size. For a specimen to represent bulk material properties, this ratio should be greater than 10-12 [2].

As a spin off, the USS specimen can also be employed for in-service life estimation of other power plant components by semi-destructively scooping out small amount of material from critical location to carve out tensile sample. USS specimen can also be employed during failure analysis, weld joint strength estimation, coating strength evaluation, exotic alloy development etc where availability of material is a constraint [3].

Prior to employing USS specimen for irradiated material testing, it has been qualified by testing on various ferrous and non ferrous alloys and by comparing the mechanical properties obtained from USS specimen with sub-size and standard ASTM specimen. Specimen design is carried out in such a way that the stress distribution during testing is similar to that of standard specimens. Specimen fabrication methods have been formulated to achieve required geometrical tolerances and surface finish. Strain measurement during testing is carried out using a video-extensometer as conventional extensometers cannot be attached to USS specimen owing to its small gage length. Thus a study carried out to qualify the USS specimen for use on irradiated material is elaborated in this paper.

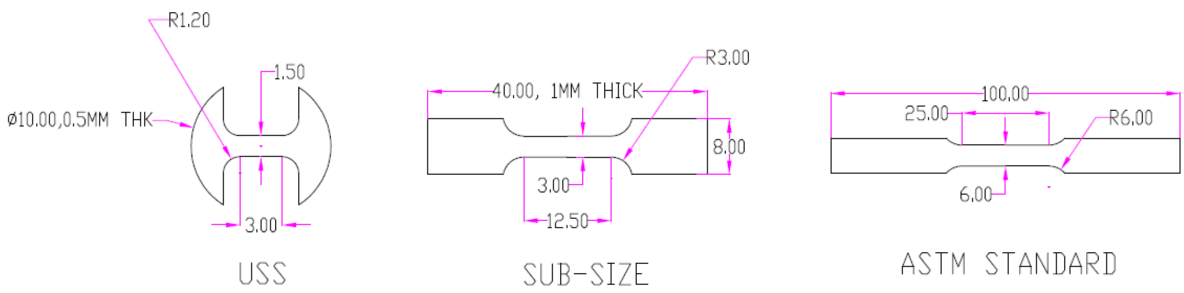

Fig 1.1 Ultra sub-size, sub-size and ASTM standard tensile specimens

2. Specimen Design

International standards such as ASTM recommend two types of geometries for a tensile specimen namely cylindrical and flat. Miniaturization has been carried out by various investigators in both the types of tensile specimen geometries mentioned. But as most of the fast reactor components are thin walled structures, it is appropriate to choose a flat specimen as it can be extracted from actual nuclear reactor components. Flat specimens have been miniaturized by employing different geometries based on suitability of manufacturing and gripping as shown in Fig. 2.1 [2]. USS specimen is a disc type tensile specimen carved out of a 10mm diameter and 0.5mm thickness disc. Disc type specimen is selected as currently disc type specimens are carved out from in service components for shear punch testing and density measurement. Also the irradiation volume available in reactor is cylindrical in shape which advocates use of a disc type specimen for efficient utilization available space. USS specimen has 3mm gage length, 1.5 mm gage width and 0.5mm thickness. Fillet radius affects stress concentration and and shold be as large as possible which in turn increases overall size of the specmn. Thus, fillet radius and overall disc diameter was optimized to vule of 1.2mm and 10mm respectirely using Finite element analysis (FEA)

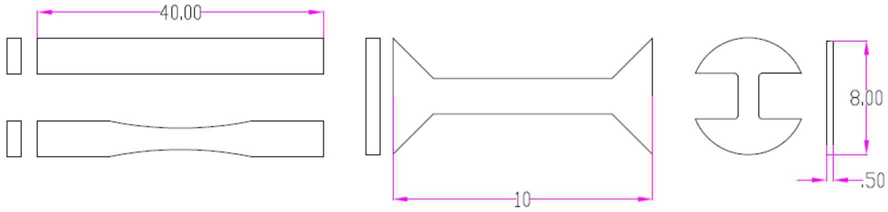

Fig 2.2 Miniaturized tensile specimen geometries attempted earlier [2]

Nominal sizes being fixed, geometrical tolerances were determined for USS specimen. ASTM E8M - Standard Test Methods for Tension Testing of Metallic Materials specifies tolerances on flat specimen as shown in Table II

TABLE II: DIMENSIONS OF TENSILE SPECIMEN GEOMETRIES AS PER ASTM E8M

Dimensions(mm)	Plate type	Sheet type	Subsize
Gage Length	200.0±0.2	50.0±0.1	25.0±0.2
Width	40.0±2.0	12.5±0.2	6.0±0.1
Thickness	Thickness of material		
Fillet Radius	25	12.5	6
Width of grip section	50	20	10

Thus for a width of 6mm a tolerance band of 0.2mm is provided which is ~1.7%. In case of sub-size sample used in IGCAR having 3mm width and 1mm thickness, a tolerance band of 0.02mm is specified which 0.67% of the width. Tolerance on USS having 1.5mm width and 0.5mm thickness is specified as tolerance band of 0.02mm based on ease of fabrication which results in tolerance of 1.33% of width. To study effect of tolerance on total elongation, FEA analysis was carried out on geometries of different sizes by varying dimensional parameters of width, thickness and length in gage region, shoulder radius for ultra sub-size as well as standard sub-size specimen. Stress-strain curve of SS316 were used to get elongation at specified load applied on the specimen so as to deform it into plastic region as close as possible to UTS point on the curve. The values of load and elongation were used for comparison with FEA carried out on geometries of USS by varying dimensions.

The stress strain curve was obtained by applying multi linear stress strain curve of SS316 to different geometries and applying force boundary condition in FEA so as to get stress value as close as possible to the UTS value specified in the multi-linear model. Initially, the USS specimen geometry with nominal dimensions was analyzed using multi-linear stress strain curve of SS316 material. From analysis, stress strain curve was generated using displacement data of gage section and average stress value at the centre of the specimen. The stress strain curve generated from FEA and that given as input was in agreement thus establishing validity of the model and boundary condition used. Load value had to be adjusted in such a way that maximum stress in the model does not exceed maximum stress value specified in the stress strain curve. Due to dimensional variation of the geometry from nominal size, it was expected to obtain deviation from the stress strain curve. Thus analysis was performed by changing parameters like gage width, thickness and fillet radius from nominal values to lower extreme of the tolerance band to study its effect on stress strain curve. Results of standard FE model in comparison with specimen having uniformly reducing width, thickness and reduced shoulder radius is shown in Fig 2.3

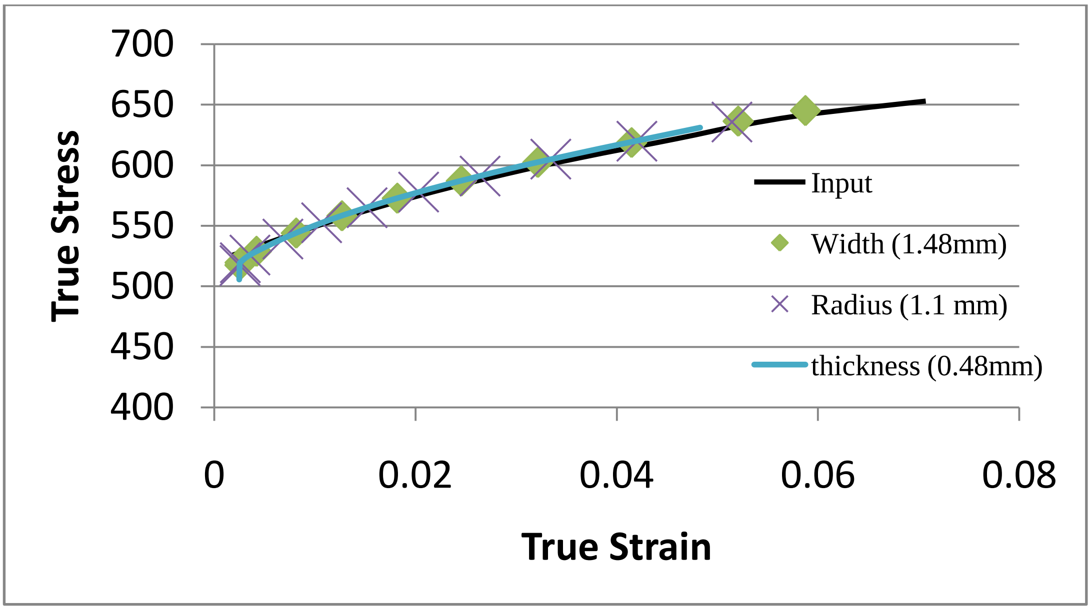

Fig 2.3 Stress Strain curve obtained from FEA of USS geometry for various tolerance values

Thus, varying the dimensions does not introduce large variation in stress strain diagram up to work hardening region. Hence 0.02mm all around profile tolerance was specified for USS specimen.

Surface roughness is another important geometrical parameter. The specimens will be fabricated using wire EDM. Removal of a recast layer created during the wire EDM process from the miniature specimen surface using a 1000 diamond grit paper eliminates the effect of specimen fabrication on base material [8]. This procedure was adopted before testing of the USS specimen.

3. Experiments

To cover a wide range of engineering materials, different types of materials were chosen for testing. Predominantly used materials in nuclear and pressure vessel industry namely austenitic and ferritic stainless steels like SS316, Mod 9Cr1Mo, RAFM were selected for testing. Non ferrous metals like copper and aluminium were opted to cover non-ferrous metals. Mar aging steel and 40% cold worked SS316 were chosen to include high strength and low ductility materials. An extremely brittle composite of titanium carbide nanotubes in titanium metal matrix was evaluated to study brittle materials. Pressure vessel carbon steel SA333 was also selected as carbon steels are most rampantly used steels. The materials were chosen based on their availability and suitability and were in the form of rectangular plates out of which specimens were extracted. As mechanical properties obtained from USS specimen had to verified, 40mm long 12.5mm gage length, 3mm gage width and 1mm thick sub-size specimen and 100mm long, 25mm gage length, 6mm gage width and 2mm thickness ASTM specimen were chosen for comparison. Four samples of each type were extracted from above mentioned materials and tested in similar conditions to get mechanical properties like yield strength, ultimate tensile strength, uniform elongation and overall elongation.

Grippers provided with UTM machine were capable for testing standard specimens. For sub-size specimen testing, grippers have been developed in RML and have been successfully employed in testing at room temperature and high temperature tensile testing of un-irradiated and irradiated specimens inside the hot-cells shown in fig 3.1(a).

For USS sample even small loads can cause bending loads in the gage section. If the gripper is bulky then there is high probability of inadvertent loads on the specimen causing erroneous results of the test. Hence sub-size sample grippers cannot be used as is for the ultra sub-size sample. A gripper was developed for ultra sub-size specimen (fig 3.1(b)). The gripper ensures axial loading of the specimen and accounts all other non-axial loads to be borne by the gripper. To maintain flatness, it was ensured that the specimen is always pressed against a flat machined surface. A small 0.25mm depth depression is provided for location of sample during loading of the specimen. This ensures verticality of the sample.

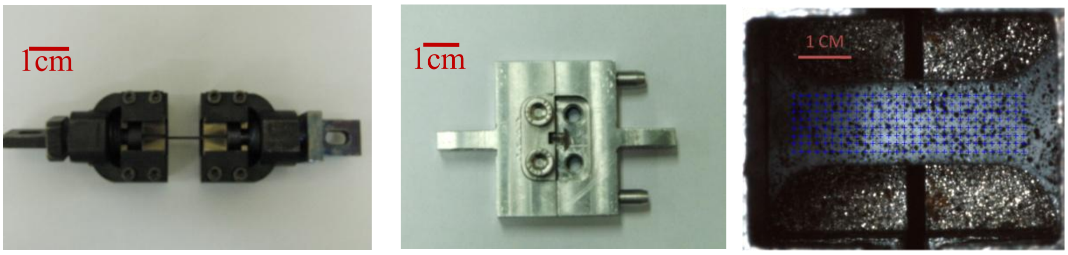

Fig 3.1: Gripper employed for a) Sub-size specimen b) USS specimen c) DIC for strain measurement

As conventional extensometers cannot be employed for strain measurement, a video-extensometer was employed and Digital image correlation was attempted. . In Digital image correlation, images of a random pattern of black dots sprayed on a white background on specimen are captured using a camera during the test (fig 3.1(c)). As specimen gets deformed pattern also deforms along with. This deformation pattern is captured during the test through pattern analysis to convert into strain distribution using image processing algorithms. This gives direct measure of strain distribution during the test [9]. DIC was used to get strain directly from the sample in the gage length and eliminate all the machine and gripper compliances. DIC also depicts elongation of fillet radius due to yielding which was predicted in FEA. Conversion of displacement data into strain will give strain map of the specimen which can be directly compared with FE model. Strain pattern is consistent with the strain pattern observed on tests previously carried out on standard samples with DIC. Thus it helps in verification of loading setup and testing procedure.

ASTM E8M standard specifies strain rate of 0.05 – 0.5 mm/mm/min. For 3mm gage length cross head speed is 0.15-1.5mm/min. All USS samples were tested at 0.2mm/min, sub-size samples were tested at 0.5mm/min and standard samples were tested at 1mm/min all at room temperature. Few tests were carried out at temperatures of 400 and 550^{o}C to see effect on stress strain curve. Tensile tests were carried out on three types of tensile specimens names USS, sub-size and ASTM standard employing ferrous and non ferrous alloys.

4. Results and discussion

The results obtained by testing of USS specimens of same material show scatter in the mecahnical properties like tensile strength, yield strength and uniform strain which is less than 5% for most of the materials tested. The scatter in the values depends on various factors like intrinsic scatter in the available material, variation in the geometry of the specimen, variation in clamping loads, uniaxiality of the load train, error in dimensional measrement and repeatability etc. The results obtained from tensile testing of USS, sub-size and ASTM standard specimens over a range of materials are shown in Table II.

TABLE II: MECHANICAL PROPERTIES FROM VARIOUS SPECIMENS

Material	**ASTM (100)**	**Subsize (40)**	**USS (10)**	**% Variation with ASTM**	**% Variation with Subsize**
SS316	-	564(±0.2%)	560(±1.2%)	-	-0.62
Mod 9Cr1Mo	675(±0.8%)	648(±0.3%)	664(±1%)	-1.63	2.47
Maraging Steel	969	-	930(±3%)	-4.02	-
RAFM	-	629	637	-	1.27
SA333	466	447	459	-1.50	2.63
Aluminum	214(±1%)	209(±2.3%)	204(±1.8%)	-4.67	-2.39
Copper	335(±0.2%)	326(±0.6%)	319(±1.8%)	-4.78	-2.15

Stress strain curves obtained from USS specimens for a typical material are shown in fig 4.1. Fig 4.2 shows stress strain curves obtained from USS specimen for a range of materials. Comparison of stress strain curves obtained from USS, subsize and ASTM standard is shown in fig 4.3 Tensile tests on ultra sub-size specimens on different materials (aluminum, copper, carbon steel, SS316, Cr-Mo steels, Maraging steel) exhibited a scatter of about 2-5% in the tensile properties. The 0.2% proof strength and ultimate tensile strength from ultra sub-size specimen tests were within 2-7% and 2-5% respectively of the sub-size/ASTM specimen results, while uniform elongation of ultra sub-size specimens were within 4-6 % of large size specimens. This study suggests that tensile tests with ultra sub-size specimens (1.5 mm gage width, 3 mm gage length and 0.5 mm thick) have the potential to provide the stress-strain data representative of bulk material and is useful not only for the nuclear industry but also for other applications like life estimation of any in-service component and for development of new and exotic materials where material availability is limited.

5. Summary

As USS specimen is found to be suitable for mechanical property evaluation of structural materials it will be implemented for testing of irradiated materials by irradiating developmental alloy specimens inside reactor and by extracting samples out of structural components of fast reactors. Further efforts are required to formulate standards through round robin testing, before the technique can be deployed in the field.

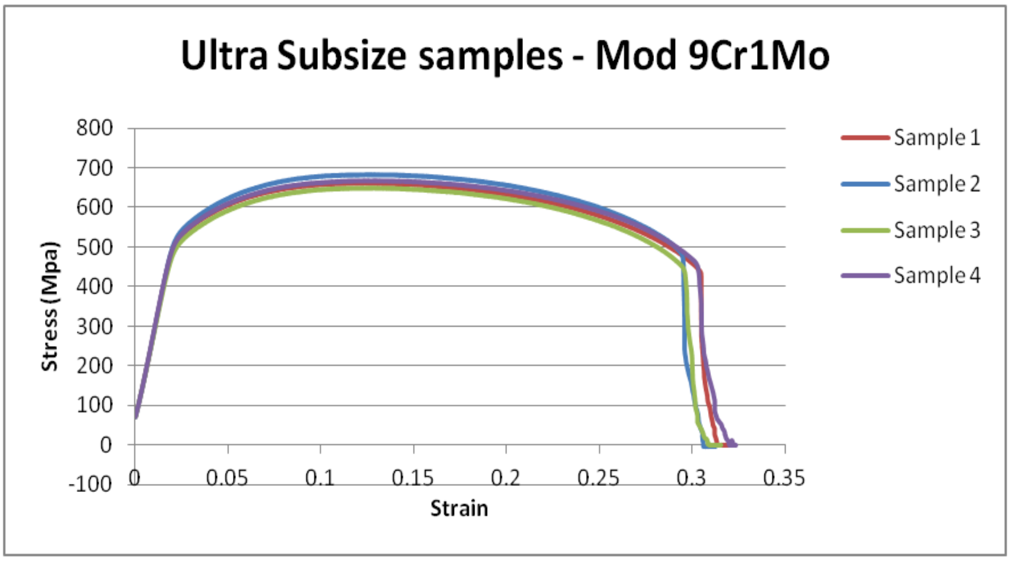

Fig 4.1: Stress strain curves obtained from USS specimen from same material

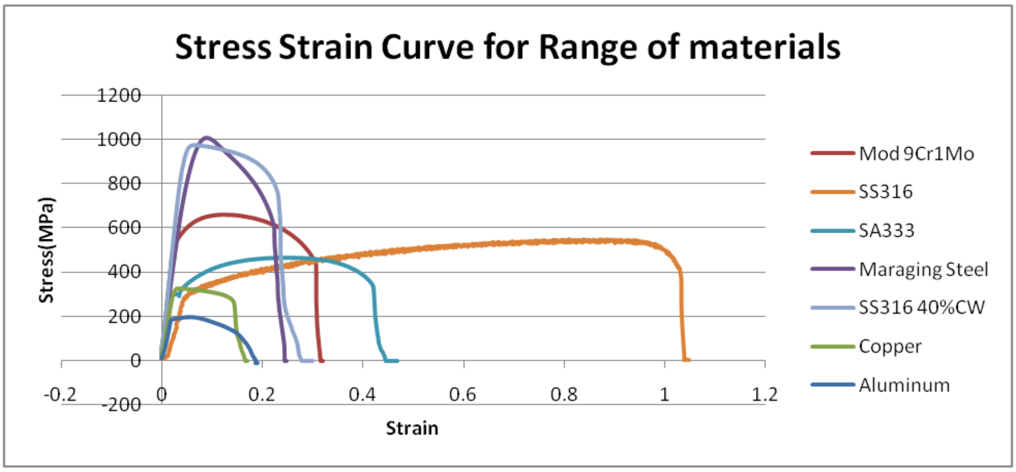

Fig 4.2: Stress strain curves for a range of materials obtained by testing USS specimen

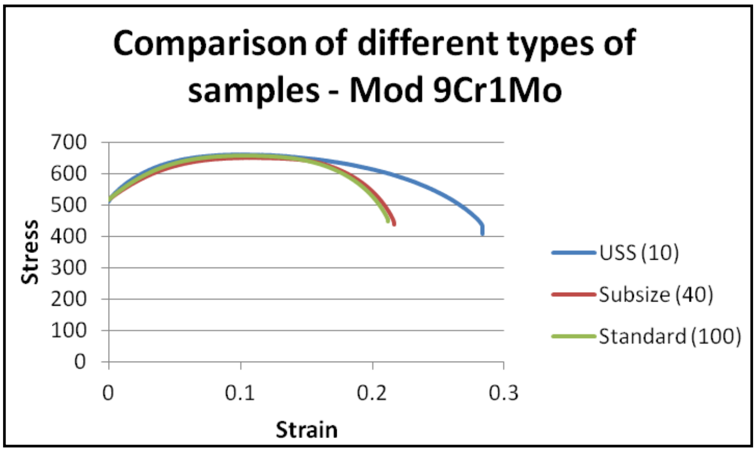

Fig 4.3: Stress strain curves obtained from three types of tensile specimens

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