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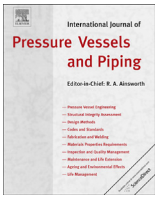

Application of small punch testing on the mechanical and microstructural characterizations of P91 steel at room temperature

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ABSTRACT

The use of small punch test (SPT) has emerged as a potential technique for mechanical characterization using miniaturized specimens. There is a strong interest in applying SPT for life prediction of power plant components operating at high temperatures. Another important application includes implementing surveillance programs for structural materials of nuclear plants where small volumes of irradiated samples are needed. In this work the small punch test was applied to study the mechanical behavior of P91 steel at room temperature. The selection criteria of the characteristic load P_Y in terms of two methods are discussed. Using this parameter the relationship with the yield stress is studied. The correlation factors were calculated from SPT curves. Microhardness was used to detect the most strained zones in a cross section of a punched specimen when reached its maximum load. The annular zone under the ball contact area, where the plastic thinning occurred up to maximum load, coincided with the highest microhardness values. Finally, transmission electron microscopy was employed to study the final microstructures after the deformation of the tensile and small punch tested samples. Refining of subgrains and considerable increasing of dislocation density was found with both, tensile and punch tested samples. By estimation of local strain and TEM observations it was confirmed the SPT produces higher deformation than that found with tensile test after rupture.

1. Introduction

The 9–12%Cr creep-resistant ferritic-martensitic steels are progressively more considered for pressure vessels, boilers, heaters and piping applications [1,2]. In particular, Grade 91 steel is widely used in the energy industry due to properties like: high creep strength, high thermal conductivity, low thermal expansion, good corrosion resistance and good mechanical properties after irradiation [3–5]. Grade 91 steel is a candidate for structural components of Generation IV nuclear power plants and future fusion reactors [1,6,7]. However, this steel could degrade its mechanical properties under thermo-mechanical cycling [8–12]. Therefore, there is an increasing demand of the accurate determination of the already mentioned properties during service. Moreover, it is also important to correlate the variation of the properties with the change of material microstructure. For this reason, miniature specimen tests are required in order to sampling without affecting the operation of the components. Furthermore, the selectivity of the extraction of the samples becomes an advantage.

The Small Punch Test (SPT) is a technique settled for evaluating mechanical behavior from miniaturized thin disks. Typically, for this type of test, two specimen sizes are used 8–10 mm [13–15]: and 3 mm [16–18] in diameter with 0.5 mm and 0.25 mm thick, respectively. In certain cases, the disk can be obtained from a small chip extracted from a component in service without affecting its performance. For this reason, the technique could be considered as a non-destructive method. For example, SPT is very useful for creep life assessment for in-service components including welded joints [2,19,20].

The SPT can be easily described as an axis symmetrical specimen punched with a ball. The typical 'applied load vs. central disk displacement' curve for ductile steels could be divided into four different regimens [16,21]: (i) elastic bending; (ii) plastic bending; (iii) plastic membrane stretching; and, (iv) plastic instability. The geometry of the test imposes on the sample a complex stress field. The equivalent plastic strain at certain locations of the punched disk can reach values much larger than the true uniform strain [18].

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The correlations between the values determined from SPT and conventional mechanical properties are still under debate nowadays [13,15,21–24]. Most of the works on Grade 91 steels by SPT have been performed at high temperature [2,12,25–27]. To the best of the author's knowledge there are no reports over Grade 91 steel that correlate the punched specimens with their deformed microstructure after SPT analyzed by transmission electron microscopy (TEM). The combination of both techniques, SPT and TEM, in other steels has been reported only by Byun et al. [28,29] using 3 mm diameter disk. However, Fan et al. [30] reported the change of the microstructure produced by equal channel angular pressing (ECAP) over T91 at room temperature. They found that the increasing number of extrusion passes, the average grain size was gradually refined. Also, the Vickers microhardness increased in 40% for the initial sample after six ECAP passes. In the work of Zhang et al. [31], dynamic plastic deformation (DPD) is applied to modified 9Cr-1Mo steel at room temperature. They correlated the improving of tensile properties, the increasing microhardness and the changes of the microstructure processed by DPD.

The aim of this work is to associate the SPT with TEM to study the changes in microstructure of P91 steel. The deformed samples are extracted from SPT specimens to be observed by TEM. The mechanical response of P91 steel is studied using SPT at room temperature. The correlation between the flow stress and the characteristic load (P_Y) based on two different methods is discussed. Aspects regarding to the top and bottom displacement measurements are analyzed and compared.

Because of the complexity of stresses during SPT, a wide range of plastic strain is found throughout the tested specimen. The variations of such strains are distributed in both radial and thickness directions. Microhardness is used over a cross section of a tested specimen to detect the highest strained locations. The local strain is studied analyzing the microstructure by transmission electron microscopy. Finally, the microstructure of (i) a normal to the symmetry axis small punch tested sample, (ii) a radial cross section of small punch tested sample, (iii) tensile tested, (iv) as received P91 steel are compared by TEM.

2. Material and methods

2.1. Material

The material under study is the ferritic-martensitic ASTM A335 grade P91 steel provided by JFE Steel Corporation, Japan. The material was delivered in the form of pipe of 355.6 mm in diameter and 28 mm in thickness. The chemical composition is given in Table 1. The heat treatment performed by the provider was normalizing at 1050 °C for 10 min, and tempering at 785 °C for 45 min. Under these thermal treatments, the obtained microstructure of the as-received (AR) material consists in tempered martensite.

2.2. Mechanical characterizations

Cylinders with a diameter of 10 mm were obtained by machining in parallel direction to the pipe axis. A single tensile test specimen was turned from the cylinder. The thinned and polished section of the tensile test specimen had a diameter equal to 5 mm. The tensile properties up to rupture were obtained using a servo-hydraulic MTS810 testing machine. The initial strain rate was 5×10^{-4} s^{-1}. The first part of the tensile test was monitored by an extensometer MTS 632.13F-20 with a gauge length of 10 mm.

For SPT specimens, slices of 0.9–1 mm in thickness were cut, using a diamond saw Bueler, from the same cylinder used for tensile test. Then mechanical grinding was performed to fit the thicknesses to 0.400, 0.500 and 0.600 mm with emery paper (320–2000 grit) and finally polished with 1 μm colloidal alumina suspension. In all cases the thickness variation was lower than 1%. For the small punch test it was used a special specimen holder, consisting of two dies that hold and clamp the disk-shape specimen (Fig. 1a). Fig. 1b shows the 2.5 mm diameter ball of silicon nitride in contact with the puncher and the dimensions details of the lower die. This equipment allows measuring the relative displacement between the punch and the upper die, by coupling an extensometer MTS 632.12C-20, with 25 mm gauge length.

In addition, the central displacement of the specimen was also monitored by measuring the displacement of the bottom face of the disk using a contact rode (Fig. 1a) coupled to a linear variable differential transducer (LVDT) HBM model W1T3 (±1 mm). The SPT specimens were tested at room temperature and using a constant displacement rate of 0.1 mm/min driven by an Instron 5567 testing machine. A load cell of 5 kN was used to record the applied load. In the case of sample with 0.500 mm in thickness several tests were carried out. Samples with 0.400 mm and 0.600 mm in thickness were tested only up to maximum load P_{MAX}.

Vickers microhardness testing was performed using a Mitutoyo MVK-H0 hardness tester. The load of 100 g was selected with a dwell time of 20 s. Three different conditions were evaluated: AR, broken tensile and small punched. The two directions of the AR material, i.e. transversal and longitudinal to the pipe axis, were indented a minimum of ten times. The same criterion was taken for the broken tensile specimen. The specimen in 0.500 mm in thickness was stopped at maximum load and it was carefully included, cut and polished up to 0.05 μm silica suspension. Microhardness scanning was performed on the half part of this cross section. Indentations were separated at least three indentation diagonal lengths in order to avoid local strain effect.

2.3. Microscopy observations

Different specimens were examined by optical microscopy (OM), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The bottom surfaces (opposite to ball contact face) of the SPT deformed specimens were observed by SEM FEI 515. Also, the cross section of the small punched 0.500 mm of thickness specimen was observed by OM Leica DMRM and SEM.

To study in detail the microstructural changes generated by the SPT, samples of AR, tensile and punch tested were examined by a TEM FEI CM200UT operated at 200 kV. In order to obtain a good correlation between the developed microstructure and the deformation induced by SPT, the identification of the region over which the observation is going to be performed is very important. Since the SP tested sample presents an axial symmetry, the plastic strain distribution varies both radially and across the thickness for the any load level of the stopped test. Therefore, it is essential to conduct a careful sample preparation having a spatial reference.

Table 1
Chemical composition of ASTM A335 grade P91 steel (1×10^3 wt%).

C	Cr	Mn	Si	P	S	Ni	Cu	Mo	Al	Co	Nb	Ti	V	Fe
107	9260	430	318	30	9	173	36	860	4.3	27	88	1.7	210	Bal.