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Small Punch Tests on Steels for Steam Power Plant (I)

— Ductile-Brittle Transition Temperature —

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Conventional test methods for measuring fracture toughness require the removal of large material samples from in-service component. However, recent developments of small punch test technique using miniature specimens have proved its usefulness and accuracy to evaluate the mechanical properties of components. Correlations have been obtained between mechanical characteristics determined from small punch test and uniaxial tensile test. Furthermore, the study showed that an appropriate empirical based-approach could be used to evaluate the Charpy-FATT of as-received and ex-service materials from small punch test.

Key Words: Small Punch Test, Tensile Test, Charpy Test, Fracture Toughness, Fracture Appearance Transition Temperature, Ductile-Brittle Transition Temperature.

1. Introduction

Accurate remnant life assessment techniques are required for steam power plant in order to avoid premature failures. These techniques are critical for the evaluation of remaining life, and establishment of life extension. Due to deviations in operational practice such as unforeseen or intentional cycling, more severe service degradation can be expected. To prevent the possibility of catastrophic failure, components are regularly inspected based essentially on the recommendations of turbine or boiler manufacturers.

With the great concern of premature failures prior to the end of design life, many utilities would like to extend the life of the power plant beyond their design life. Therefore, determination of accurate mechanical properties are necessary to increase components reliability, to optimize operating procedures and inspection intervals, as well as to define a guide maintenance and repair strategies for extending component life.

Several methods can be used to evaluate the degree of degradation induced by thermal aging. These methods are based on measuring the variation of magnetic, chemical or ultrasonic properties. Service degradation can be correlated to modification of physical properties as well as mechanical properties (Viswanathan *et al.*, 1993).

Among mechanical characteristics, fracture toughness, K_{IC}, is considered to be a key factor in the prevention of catastrophic crack growth. Knowledge of the evolution of the fracture toughness is therefore required, especially in order to avoid brittle fracture. Many studies are still focused on the determination of correlations between fracture toughness and ductile-brittle transition temperature (DBTT) (Iwadate *et al.*, 1994). The available fracture toughness data of metals are mainly provided from Charpy V-notch (CVN) impact tests. Unfortunately, the significant volume of sample material required for conventional measurement of the fracture toughness and its evolution with temperature implies the removal of large material samples from in-service components.

Exploratory research on the used of a punch-and-die mechanical test configuration was driven by the demand to assess material properties from miniature sized specimens. In an early work, Lucas *et al.* (1986) have investigated ways of extracting strength and ductility from the load-

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Table 1 Chemical composition of the different steels.

Composition	C	Si	Mn	P	S	Ni	Cr	Mo	Cu	Al	W	V	Pb
1Cr-0.5Mo	0.18	0.25	0.7	0.015	0.016	0.1	0.93	0.45	0.1	0.006	—	—	—
2.25Cr-1Mo	0.15	0.4	0.43	—	—	—	0.25	0.01	0.2	0.008	0.1	0.02	0.11
12Cr-1Mo	0.21	0.34	0.59	0.018	0.004	0.61	11.5	0.85	—	—	0.05	0.24	—

displacement curves obtained from small punch tests, and examined the sensitivity of the data to various test parameters. They observed that the load displacement behavior varied with the ball diameter, *d*, hole size, *D*, and specimen thickness, *t*, in such way that the maximum load increased and ductility decreased with d, while the yield load was only dependent on *t*.

Several authors observed that the small punch test successfully produced well defined ductile-brittle transition-temperature (DBTT) characteristic and demonstrated a significant relationship between the small punch test transition temperature and the conventional Charpy fracture appearance transition temperature (FATT) (Baik *et al.*, 1986, Mao et al., 1987, Suzuki *et al.*, 1993, Foulds *et al.*, 1994).

Consequently, this investigation was undertaken to assess the small punch test load deflection behavior, the ductile-brittle transition temperature, and the evolution of the transition temperature in ex-service components.

2. Materials and Experimental Techniques

Small punch tests have been performed on steels for steam power plant applications. The chemical compositions, in wt. %, are reported in Table 1.Specimens were machined from parts of new (as-received) or used (ex-service) superheater/reheater headers. Tables 2 and 3 are summarizing, respectively, the heat- treatments and service operations experienced by the headers.

Our small punch test device consists of a lower die, upper die, ball and punch, as described in Fig. 1.Specimens are 10 × 10mm and 0.5mm thickness with a 1 μm thickness tolerance. The displacement of the punch was obtained by placing the small punch test device on an electro-mechanical Instron tensile testing machine. Constant displacement rate of 0.25mm/min was imposed until a 20% load decrease occurred.

Table 2 Heat-treatment conditions of the studied materials.

	1Cr-0.5Mo	2.25Cr-1Mo	12Cr-1Mo
Temperature	680° ± 20°C	720° ± 20°C	745° ± 15°C
Holding time	1h/inch	1h/inch	1h/inch
cooling	furnace	furnace	furnace

Table 3 Service conditions.

Steel	Pressure	Temperature	Time
1Cr-0.5Mo	100 kg/cm^2	515°C	143.000 h
2.25Cr-1Mo	195 kg/cm^2	502°C	86.202 h
12Cr-1Mo	190 kg/cm^2	540°C	22.125 h

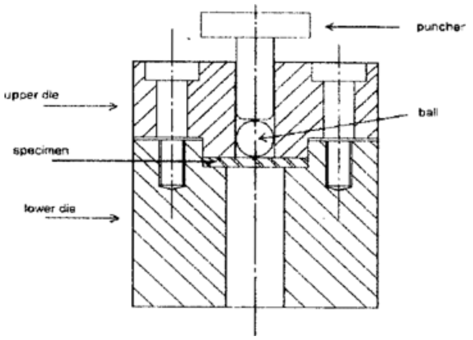

Fig. 1 Sketch of the small punch test device.

High temperature experiments were performed using an induction heating device, with the coil positioned around the small punch test jig. A special equipment has been designed for the investigation in the low temperature range. For this purpose, the small punch test device was placed in a circular chamber cooled by a circulation of liquid nitrogen in a copper tube coil. The liquid

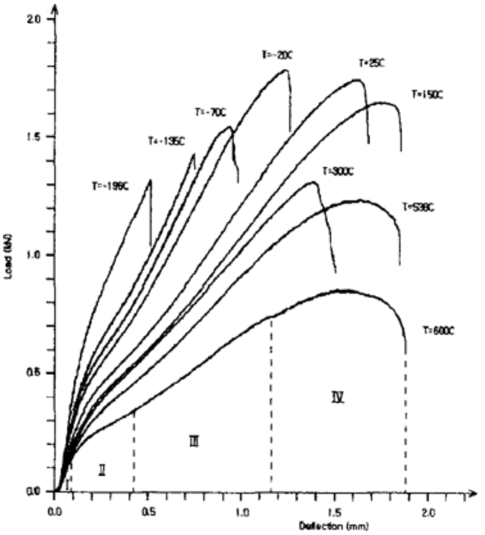

Fig. 2 Load-deflection curves on 12Cr-1Mo.

Table 4 Unixaial tensile test characteristics.

		25°C	150°C	300°C	420°C	538°C	600°C
1Cr-0.5Mo	σ_y (MPa)	304	—	278	255	230	—
	UTS (MPa)	464	—	480	473	392	—
	el. (%)	29.6	—	19.7	23.5	28.6	—
2.25Cr-1Mo	σ_y (MPa)	310	—	285	260	235	—
	UTS (MPa)	520	—	504	485	392	—
	el. (%)	33.3	—	21.5	25.3	28.9	—
12Cr-1Mo	σ_y (MPa)	435	410	375	355	310	250
	UTS (MPa)	752	654	604	566	449	367
	el. (%)	23.1	21.2	20.8	16.0	22.4	29.2

nitrogen was introduced by the pressure building naturally within the vessel.

The reading of temperature was made by a thermocouple type K (T>25°C) or type T (T<25°C) spot-welded on the lower die at approximately 2mm from the specimen.

3. Results and Discussion

3.1 Small punch test load-deflection

As previously described by Baik et al. (1986) the small punch load-deflection response can be partitioned into four regimes corresponding to: (I) elastic bending deformation associated with local surface micro-yielding, (II) plastic bending deformation, (III) membrane stretching, and (IV) plastic instability (Fig. 2). In the high temperature range, failure is associated with the formation of necking, or local deformation, as it is observed in tensile test performed on ductile materials. With decreasing temperature, the nature of failure changes. At low temperature, an early crack initiation and rapid crack growth within the plasticity regime are resulting to a brittle failure before having reached the membrane stretching and plastic instability regimes.

3.2 Comparison with tensile test

In a first approach, we will qualitatively compare values measured from small punch tests with mechanical characteristics obtained from uniaxial tensile tests. For this purpose, we used tensile test data published by Yoon (1985) on 1Cr-0.5Mo and 2.25Cr-1Mo steels, and data of Yoo Jin (1996) for 12Cr-1Mo steel. The temperature dependence on the mechanical properties are reported in Table 4, with the yield stress, σ_y, and ultimate tensile strength, UTS, decreasing when the temperature is increasing, while the ductility, or elongation el., is minimal around, approximatively, 300°C.

Similar trends for the temperature dependence on the mechanical characteristics measured in the small punch tests can be observed in Fig. 2, with a decrease in the maximum load with the temperature, and a minimum value of the deflection reached around 300°C. Values of the maximum load and maximum deflection obtained from small punch tests have been reported in Fig. 3 and Fig. 4 versus, respectively, the ultimate tensile strength and elongation determined at identical temperatures from uniaxial tensile tests. As mentioned by several authors (Lucas *et al.*, 1990, Eto *et al.*, 1993, Ha *et al.*, 1997), reasonable correlations can be found, especially when comparing the maximum deflection and elongation. Yet, a larger scatter-band is obtained when comparing maximum load in small punch test with ultimate tensile strength in the particular case of 12Cr-1Mo steel. At this point of the study, it is difficult to explain this wider scatter-band.

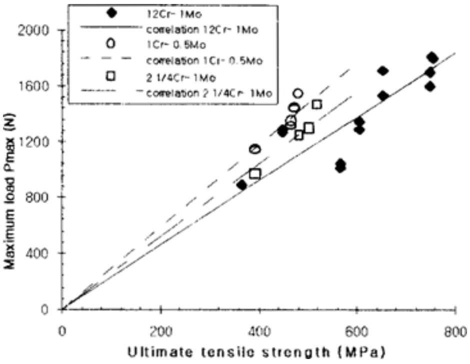

Fig. 3 Comparison between SP maximum load and ultimate tensile strength.

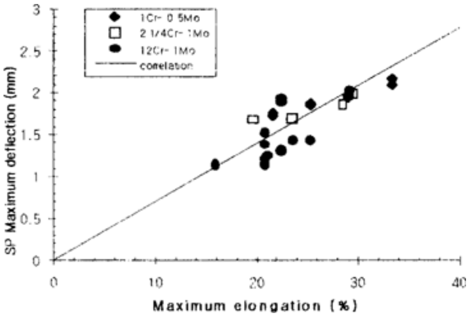

Fig. 4 Comparison between SP maximum deflection and UT maximum elongation.

However, it is expected that the grain size might be a major factor.

With a grain size of 50 μm, in comparison with 15 μm and 20 μm for 1Cr-0.5Mo and 2.25Cr-1Mo steels, respectively, the number of grains within the thickness is small, and therefore, the material behavior is expected to be affected, since the material cannot be considered as fully isotropic. Furthermore, with the formation of necking and a multiaxial stress state present within a small punch specimen, the fracture toughness might also be affected by this non-isotopic behavior.

The first regime of the small punch load-deflection curve is corresponding to the elastic deformation within the specimen. Correlations between small punch test and uniaxial tensile test mechanical properties are more likely to exist within a domain which involved small and elastic deformation.

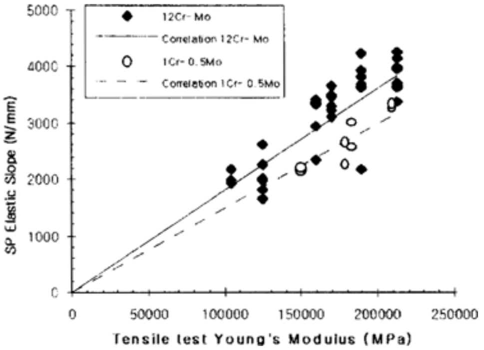

Fig. 5 Correlation between slope in SP elastic regime and Young's Modulus.

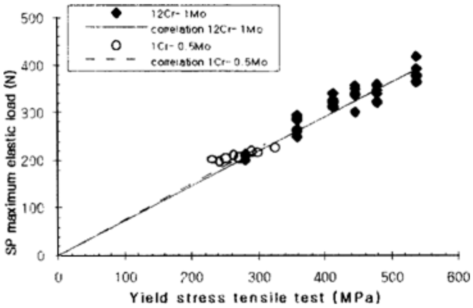

Fig. 6 Correlation between maximum load measured in the SP elastic domain with the material UT yield stress.

Slopes measured in the elastic regime and values the load measured at the end of the elastic domain on small punch test curves have been reported in Fig. 5 and Fig. 6 and compared, respectively, to the Young's Modulus and the yield stress determined from uniaxial tensile tests. The linear relations, observed between small punch test and tensile test characteristics, suggest that mechanical properties can be evaluated from small punch tests, on miniature specimen. These correlations were encouraging to model the small punch load-deflection behavior. Using constitutive equations determined from conventional uniaxial tensile test, formulations have been proposed for the modeling of the small punch test curves in the temperature range 25°-600°C (Fleury et al, 1998).

3.3 Ductile-brittle temperature transition

The main use and interpretation of the small punch test is to assess the key material fracture properties FATT and K_{IC}. It has been conclusively shown that steels, exhibiting a standard Charpy impact ductile-to-brittle fracture transition behavior with decreasing test temperature, also show a small punch test ductile-to-brittle energy transition behavior with decreasing temperature (Baik *et al.*, 1986). In the case of small punch test, the fracture energy is defined as the energy absorbed up the maximum load in the load-deflection curve.

In the attempt to determine the ductile brittle transition temperature and to establish a correlation with the FATT as determined from Charpy impact test, small punch test experiments have been performed in the temperature range −196°C to 25°C.

In the upper-shelf domain, the fracture occurred in the plastic instability regime, in the lower-shelf domain, the fracture occurred in the plastic bending regime, whereas fracture in the ductile-brittle transition regime occurred in the membrane stretching regime.

Fracture energies obtained from these small punch tests have been reported versus the test temperatures in Fig. 7 and Fig. 8 for 12Cr-1Mo steel and 1Cr-0.5Mo steel, respectively. A clear transition between ductile-to-brittle behavior can be observed associated with the fracture energy change.

On 1Cr-0.5Mo ferritic steel, a large variation of the fracture energy with temperature was observed. From 25°C, the fracture energy is first increasing when the temperature decreases to reach a maximum value around −140°C. Then, as the temperature decreases further, the fracture energy decreases significantly, showing a marked transition between ductile to brittle behavior.

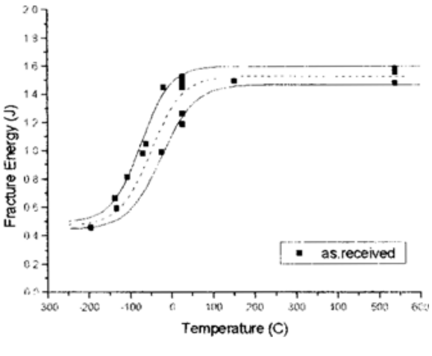

Fig. 7 Evolution of the fracture energy versus temperature for 12Cr-1Mo steel.

This evolution of the fracture energy is resulting from a modification of the crack initiation and crack propagation modes. Failure in the ductile regime occurred by cracking within the necking located along the circumferential edge of the punch. Ductile tearing region can be observed on the fracture surface with void formations (Fig. 9). When the temperature is decreasing, less plasticity is involved in the deformation, and mechanisms of crack initiation and propagation changed. In the low temperature range, cracks were initiating closer to the center of the specimen, and were following a straight and narrow crack propagation path. The resulting fracture surfaces are exhibiting transgranular cleavages.

When comparing ex-service and as-received materials, difference in the ductile-brittle transition temperature is observed, resulting from the

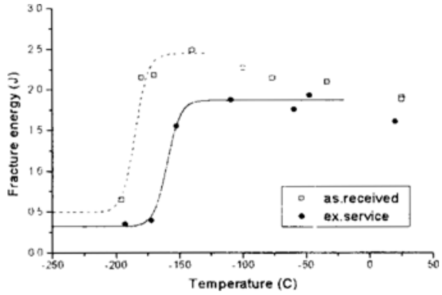

Fig. 8 Evolution of the fracture energy versus temperature on 1Cr-0.5Mo steel.

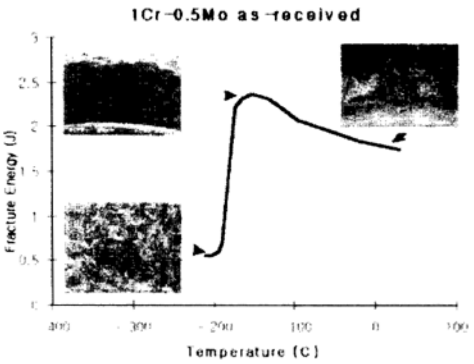

Fig. 9 SEM micrographs of the fracture surface at several temperatures and the corresponding fracture energies.

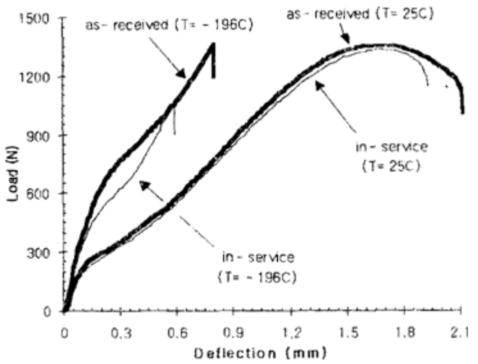

Fig. 10 Comparison between load-deflection responses on 1Cr-0.5Mo steel in as-received and ex-service conditions.

development of damage in operating components. But, for both conditions, the variation of the fracture energy from the upper-shelf energy to lower-shelf energy with decrease in the temperature from 25°C and -196°C, suggests that mechanisms controlling the cracks initiation and propagation in the ductile and brittle domains are not identical. Further metallographic observations should be performed in order to establish correlation between microstructure evolution and changes of the crack mechanisms. Nevertheless, the aging condition is expected to accentuate the formation of void and to facilitate the intergranular crack propagation in the ductile regime, affecting the crack propagation resistance and therefore the fracture toughness.

As illustrated in Fig. 10, the difference in behavior is mainly resulting from a reduction of the maximum deflection in ex-service materials in comparison with as-received. When tests are performed at the temperature above the DBTT, if ex-service materials exhibit a lower maximum deflection, the maximum load remains almost identical to as-received materials. Higher crack propagation rate in the ex-service material is suspected to be the cause of this difference in behavior, and to the lower values of the fracture energy.

The difference in behavior between as-received and ex-service materials is more pronounced in the brittle regime, as shown in Fig. 10.As suggested earlier, metallographic observations should be performed in order to identify factors and/or mechanisms responsible of this difference in behavior.

As mentioned by Baik *et al.* (1986), an analogy between small punch test can be made with Charpy test if considering the values of the fracture energy. But a more precipitous fracture energy transition behavior is obtained in the case of small punch test. A small punch transition temperature (T_{sp}) can be defined as the temperature characterizing the mid-energy of the small punch test, corresponding to the mean value between the upper- and lower-shelf. Comparing small punch transition temperatures (T_{sp}) and Charpy FATT on different alloys, several authors (Masushita et al. (1991), Suzuki et al. (1993) and Foulds et al. (1994)) have already obtained the ratio:

$$\frac{T_{sp}}{FATT}=0.35 \qquad (1)$$

where T_{sp} and FATT are expressed in Kelvin.

This shift of the ductile-brittle transition temperature (DBTT) toward lower temperature is attributed to the lower strain rate involved in the deformation and rupture of the specimen during small punch test. While almost no effect of the strain rate was detected in the ductile regime (Ha *et al.*, 1996), it is expected that higher strain rates promote brittle cracking over a wider temperature range. Baik *et al.* (1986) studied the strain rate effect in small punch tests on Ni-Cr steels and showed that a hundredfold increase of the displacement rate leads to 20% increase of the DBTT.

Charpy impact tests were performed on identical materials by the Korea Heavy Industries and Construction (Choi *et al.*, 1997). These results have been reported in Table 5, where FATT is the transition temperature for a fracture appearance corresponding to 50% ductile-50% cleavage, and EDBTT is the transition temperature obtained from the energy versus temperature curve.

Comparing these Charpy test values with the fracture energy transition temperature obtained in this study from small punch test, we obtained the following ratios:

Table 5 Charpy test DBTT on 1Cr-0.5Mo steel (from Choi et al., 1997).

	as-received	ex-service
FATT	60°C	18°C
EDBTT	70°C	27°C

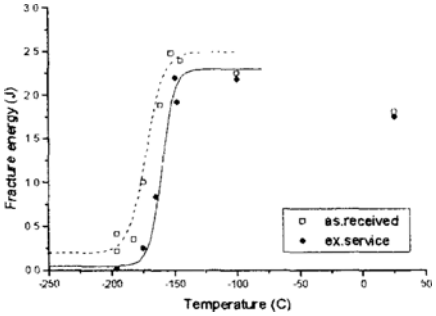

Fig. 11 Evolution of the fracture energy vs. temperature on 2.25Cr-1Mo steel.

as-received material: $T_{sp}/FATT=0.336$,
$T_{sp}/EDBTT=0.326$,
ex-service material: $T_{sp}/FATT=0.354$,
$T_{sp}/EDBTT=0.343$.

Therefore our results are found to be compatible with the relationship previously obtained in the literature.

If comparing as-received and ex-service materials, a slight shift of the FATT of about 25oC has been measured, showing the influence of material damage on mechanical properties under the combination of pressure and high temperature exposition. But, this value of the DBTT shift obtained from small punch test is lower than the FATT shift determined from Charpy test on identical material in respect to the ratio 0.35.

The small punch test behavior of the ferritic 2.25Cr-1Mo steel has also been studied on as-received and ex-service conditions (Fig. 11). The correlation obtained above from the study on 1Cr-0.5Mo steel can be used to evaluate the FATT of other types of alloy. Considering a ratio $T_{sp}/FATT=0.35$, the 2.25Cr-1Mo steel FATTs were evaluated as:

as-received material: $(FATT)_{as\text{-}received}=22°C$,
ex-service material: $(FATT)_{ex\ service}=64°C$.

These values are comparable, but slightly higher, yet, than Charpy FATT as for example, given by Suzuki et al. (1993).

4. Conclusions

In the aim to assess mechanical properties required for the evaluation of the remaining life of components used in aged fossil power plant, small punch test experiments have been performed from −196° to 600°C on a class of materials used in steam power plant.

Linear relations were obtained between mechanical characteristics determined from small punch test and uniaxial tensile test. These correlations proved the practical interest provided by the small punch test in order to evaluate the material mechanical properties from miniature specimens. Our experiments on different steels also show the effect of the grain size, which limits the application of the small punch test to material with small grains.

By conducting tests at different temperatures, a curve of absorbed energy versus temperature could be developed which is similar in shape to that of a Charpy energy versus temperature curve. The mid point of the energy curve was used to define a fracture energy transition temperature T_{sp}. Comparison with results of Charpy impact tests showed that the small punch ductile-brittle transition temperature is shifted laterally toward lower temperatures. A ratio $T_{sp}/FATT$ of about 0.35 was found on ferritic 1Cr-0.5Mo steel. This ratio lower than unity, and the mark transition between ductile to brittle regimes constitute serious disadvantages for the small punch test since the determination of transition temperature appears more delicate than from standard Charpy impact test.

This investigation also showed that aging condition resulted in a sensible increase in the fracture appearance transition temperature. The measured shift of the temperature transition was consistent with determination made from Charpy test. However, a complete optimization of the small punch test can be achieved only after a thorough metallographic study, in the aim to understand the influence of the microstructure and its evolution.

Nevertheless, these results revealed the potentiality of this technique to evaluate mechanical characteristics as yield stress, UTS and FATT values from a small volume of material which can easily be removed from components without affecting its integrity and making this test a semi-destructive evaluation method.

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