

MONITORING OF THE DUCTILE TO BRITTLE TRANSITION TEMPERATURE OF REACTOR PRESSURE VESSEL STEELS BY MEANS OF SMALL SPECIMENS

Arnaldo H. P. Andrade¹, Carlos A. J. Miranda¹ and Raquel M. Lobo¹

¹ Instituto de Pesquisas Energéticas e Nucleares (IPEN / CNEN - SP)
Av. Professor Lineu Prestes 2242
05508-000 São Paulo, SP, Brazil
aandrade@ipen.br, cmiranda@ipen.br, rmlobo@ipen.br

ABSTRACT

Neutron irradiation in nuclear power plants (NPPs) lead to microstructural changes in structural materials which induce a shift of the ductile to brittle transition temperature (DBTT) towards higher temperatures. Monitoring of the DBTT in NPP components receives therefore considerable attention. Small specimen testing techniques are developed for characterizing structural components with a limited amount of materials. One of the most used of these miniature testing is the small punch test (SPT) which is based on disc or square shaped specimens. SPTs may be performed from room to cryogenic temperatures, plotting the absorbed energy until rupture, against the test temperature. A ductile region (high energy) and a brittle region (low energy) with a transition between both zones are usually reported. The transition temperature thus obtained, $DBTT_{SPT}$, is also related through empirical expressions to the transition temperature obtained in CVN tests, $DBTT_{CVN}$, or in fracture toughness testing. Linear expressions such as $DBTT_{SPT} = \alpha DBTT_{CVN}$ have been used where α is a material characteristic constant. In all cases, the $DBTT_{SPT}$ temperature is much lower than that obtained in the CVN tests. In this paper, we present a short review of the literature on the determination of the DBTT for nuclear reactors pressure vessels steels by those two techniques analyzing the reason for the difference in their value as mentioned before. In dealing with irradiated materials, is a high priority to limit the exposure of the professional to irradiation. Therefore, the use of miniature specimens receives significant attention in the nuclear community. The high cost of irradiation experiments is a further incentive for using small specimen testing techniques.

1. INTRODUCTION

In a nuclear power plant (NPP), materials are exposed to irradiation, which can lead to embrittlement, among other effects. In order to guarantee the operation under safe conditions and perform their assessment, an accurate characterization of these effects is critical.

Radiation embrittlement of Reactor Pressure Vessels (RPV) materials is reported to consist of (a) direct matrix damage by radiation, due to high energy neutrons, (b) irradiation induced precipitation (Cu-rich) and (c) elemental segregation (primarily phosphorus). There are two major types of low alloy steels that are most commonly used for construction of RPVs: Mn-Ni-Mo steels (referred to as western steels) and Cr-Mo-V steels (referred to as eastern steels) [1].

The residual lifetime assessment and potential for possible failure of in-service nuclear components is a critical issue in the safety and reliability analysis of NPPs that are approaching the end of their design lives. A component's residual lifetime can be evaluated with the traditional and well standardized mechanical test techniques, such as the uniaxial creep, the tensile test, the Impact Charpy test or the compact tension fracture toughness test. In the circumstance of nuclear plant life extension the available material may be insufficient to extract the number of specimens required to the test program.

Embrittlement in NPPs is monitored by using dedicated Charpy specimens installed at well-defined locations in the vessel (Fig.1) [2], and having been exposed to the same irradiation and thermal conditions as the actual reactor components. In the context of lifetime extension of NPPs these specimens have become an invaluable asset.

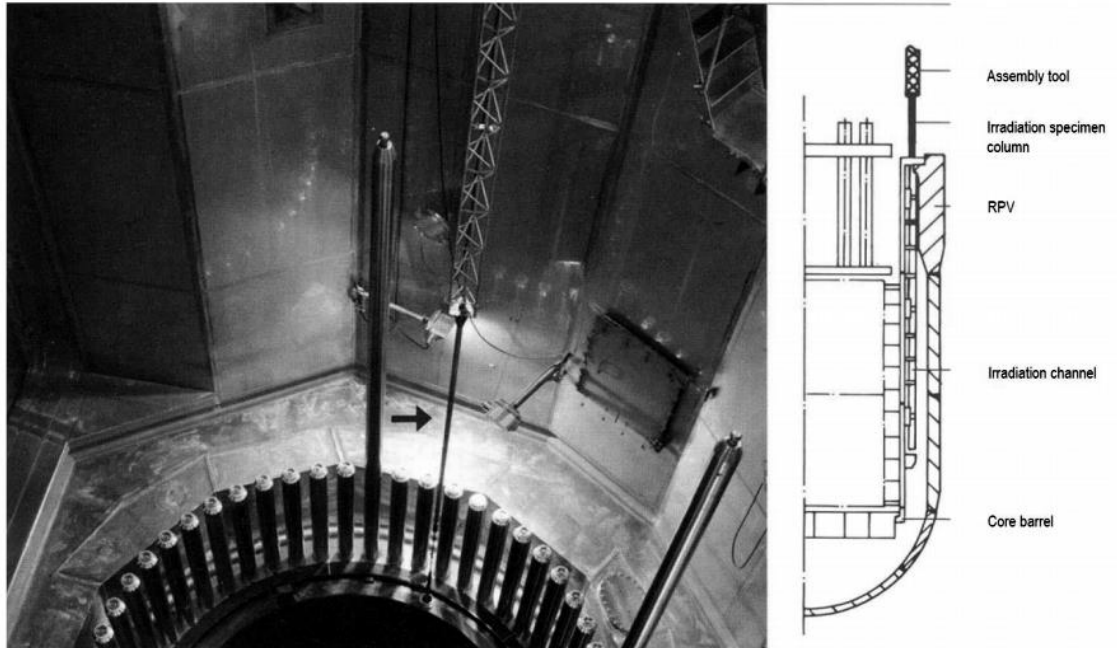


Figure 1: Surveillance Program and RPV [2]

Residual lifetime assessment is unthinkable without the knowledge of:

1. Mechanical properties of materials prior to operation, reporting all technological operations realized throughout the manufacturing of the component, and
2. Mechanical properties after actual time of operation (actual mechanical properties), because the material properties can be reduced throughout the service life by aging, service loading and temper, and hydrogen and/or radiation embrittlement.

The conventional testing methods for evaluation of the degradation and estimation of residual life of RPVs, involve standard mechanical test techniques that are direct and reliable. Though these tests can be done successfully in the RPVs fabrication stage, difficulty in extraction of the samples from the nuclear plants in service pose a serious restriction in use of these methodologies to estimate aging induce degradation for purpose of unit extension life. Thus, estimation of the mechanical properties by innovative direct testing methods led to the development of the testing techniques based on the miniaturized testing samples (Fig.2).

During the investigation of irradiated materials from fission and fusion programs limiting the exposure of the experimentalists to irradiation is a high priority. Consequently, the use of miniature specimens receives significant attention in the nuclear community. The high cost of irradiation experiments is a further incentive for using small specimen testing techniques.

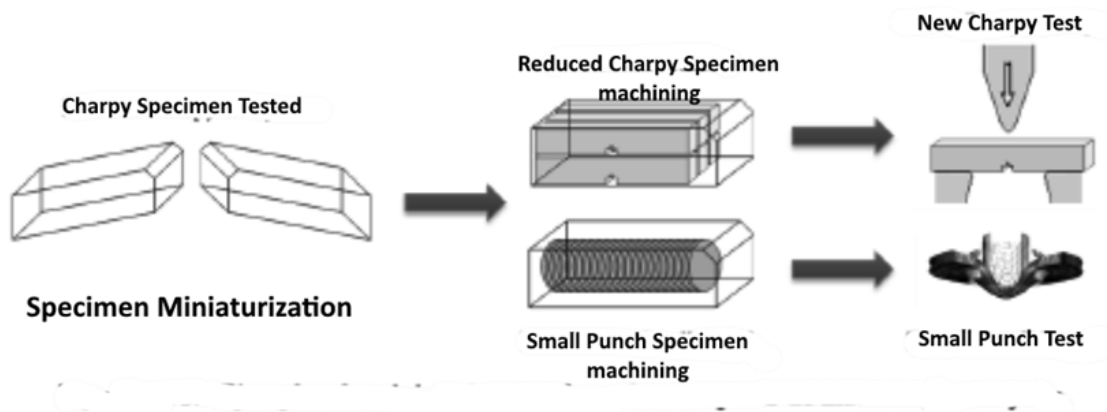
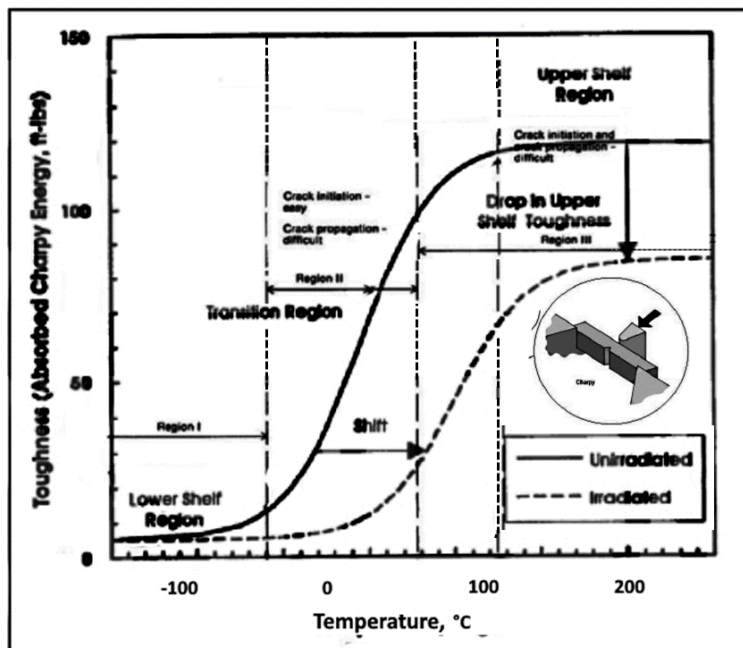


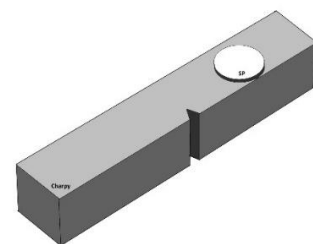
Figure 2: Specimen Miniaturization

2. DUCTILE-TO-BRITTLE TRANSITION TEMPERATURE

One of the drivers leading to the development of the small specimen testing technique was the determination of the Ductile to Brittle Transition Temperature (DBTT) and its shift to lower temperatures and neutron irradiation [3]. The standard method for determining DBTT is by means of Charpy impact testing on notched specimens with the dimensions (10x10x55 mm³) (Fig. 3a).



(a)



(b)

Figure 3: (a) DBTT Curve; (b) Comparison between CVN and Small Punch Specimens

3. THE SMALL PUNCH TEST TECHNIQUE

The need for very small specimen for testing is more useful for irradiated materials. The use of small punch (SP) test (Fig. 3b), developed in 1980s by Manahan et al [4] and Foulds [5] has emerged as a promising alternative. It is an efficient and cost-effective technique. However, there is no international standard presently. A European Code of Practice, CWA 15627, on small punch tensile and fracture tests, published by CEN (European committee for standardization) in 2006, which was further revised in 2007 [6], is being followed as code of practice.

The main elements of the Code cover the apparatus, the test specimen preparation, the test temperature considerations, the test procedure, the post-test examination, and the approaches to the derivation of yield strength (YS), ultimate tensile strength (UTS), fracture appearance transition temperature (FATT), and fracture toughness from SP tests results.

It was decided in Europe to develop the Code of Practice into a fully-fledged European Norm (EN) testing standard. It is not intended that the European standard will be developed without considering other activities worldwide. The availability of the English translation of the Standard for Small Punch Creep Test of the Japanese Society of Materials Science is eagerly awaited. In addition, both the Chinese standard announced at SSTT-2014 and the American ASTM WK47431 (New Practice for Small Punch Test Method for Metallic Materials) are expected to play a significant role in shaping the European standard.

Small Punch tests was also used to evaluate the Ductile to Brittle Transition Temperature (DBTT)/Fracture Appearance Transition Temperature (FATT) [7]. CWA 15627 Part B describes the recommended procedures for estimation of the material properties (YS, UTS, DBTT, fracture toughness etc). The SPT is basically a punch-in-a-die loading test method wherein a small, flat specimen is punched with a hemi-spherical head (punch) or a ball. The SP test for measurement of DBTT is a displacement-controlled test, i.e., the punch is pushed at a constant velocity (of the crosshead) against the specimen and the load (L) required to keep the punch moving at the constant velocity is measured as a function of displacement (D) (Fig.4).

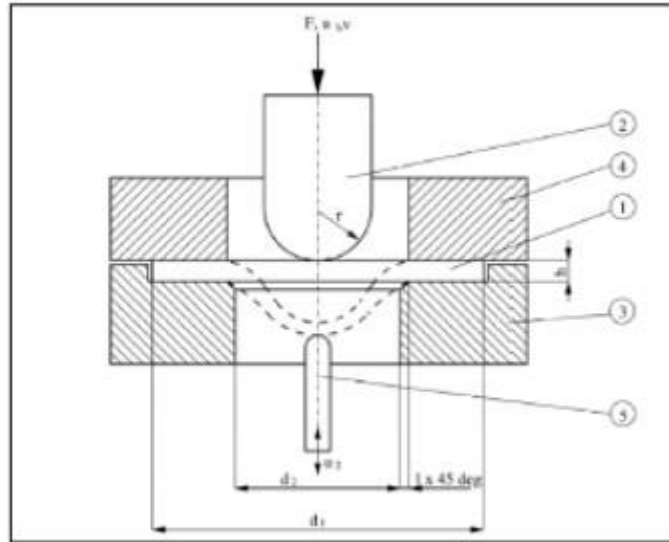


Figure 4: Scheme of Small Punch Testing Apparatus: (1) specimen; (2) punch; (3) receiving die; (4) clamping die; (5) deflection measurement rod [6]

Two types of specimen dimensions are specified in CWA 15627 Part B [b]: a frequently used “standard” specimen of 0.5 mm thickness and a “miniature” transmission electron microscopy (TEM) sized specimen of 0.25 mm thickness. Such a “standard” specimen (0.5 mm thick) is recommended because this specimen thickness is intended to assure that the number of grains through the thickness are adequate to permit bulk properties to be obtained.

The typical result of an SP test is the load-displacement (L-D) curve (**Fig.5**), showing the characteristic points which contains the information regarding the elastic & plastic deformation and the mechanical properties of the material. The DBTT is determined only from the results of SP test from empirical correlations between these results and the results of standardized tests. The correlations are highly dependent on the type of material [8]

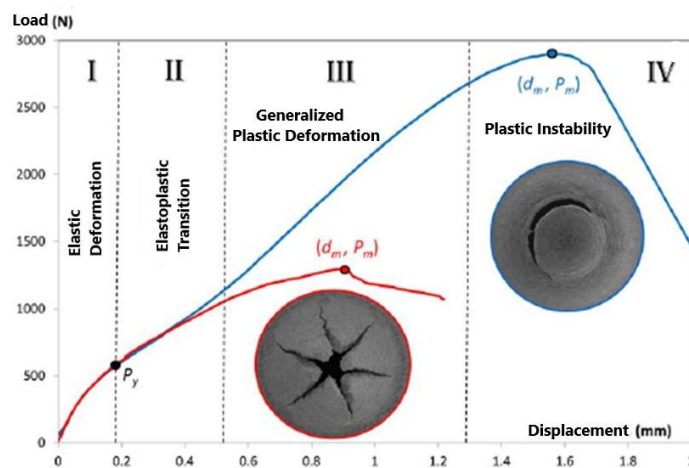


Figure 5: SP Tests characteristic curves: Blue – Ductile Iron; Red – Brittle Iron [8]

The force-deflection curve is generally divided in different stages: zone I corresponds to the indenting of the specimen surface and elastic bending. During zone II plastic bending spreads through the specimen. In zone III the specimen behavior is dominated by membrane stretching and in zone IV by necking, crack initiation, softening and final fracture.

3.1 Risks associated with the SPT

The SPT risks and uncertainties are listed in Table I and have consequences if ignored during testing [9]:

Table I: Risks involved in using SPT [9]

Types of Risk	Definition	Consequences
Safety	The SPT is not yet standardized and can be questioned as to whether it can satisfy design code requirements especially when dealing with critical components such as a nuclear reactor pressure vessel (RPV)	Public Health and Human Safety Plant Component Safety
Technical	Conversion of small specimen data to large specimens is a technical risk that has data scatter. The technique has been widely researched and improvements have been made over the years.	Plant Component Safety
Regulatory	Correlation of data to estimate material fracture toughness may not be enough to satisfy regulatory bodies as a prevention method to brittle fracture especially in a component like nuclear RPV	License issues to resolve
Economical	Safety, technical and regulatory risk will require mitigation in order to minimize all these risks, which can be costly. It may, however, look as if the SPT benefits are cancelled out by mitigation cost.	Mitigation cost can be high especially with a technology or technique that is not formally standardized since the data can be interpreted differently

For the determination of an SP tensile test data several characteristic values determined from the force-deflection $P(d)$ curve are used (**Fig.5**):

- P_m , the maximum force,
- d_m , the deflection at maximum force,
- P_y , the elastic-plastic transition force,

- E_{frac} , the fracture energy E_{frac}

To calculate E_{frac} the force P is integrated over the deflection d up to the point d_{frac} where fracture occurs (Eq.1). Different approaches for defining d_{frac} are found in literature.

$$E_{frac} = \int_0^{d_{frac}} P(d)d(d) \quad (1)$$

These definitions of d_{frac} are well established in the case of ductile fracture with smooth force-deflection curves like the one in **Fig.5**. In the case of brittle fracture, the situation is more complex. Fig. 6 shows an example of brittle failure where different drops of the force occur before the global maximum force is reached. These force drops are indicative for crack initialization [10].

The multiple load drops that are observed from time to time at load-punch displacement curve at the SP tests carried out at lower temperatures have evidenced that the occurrence of the first load drop is a consequence of the initiation of the first circumferential crack with the following load drop associated with the propagation of further radial cracks (Fig.6). In such cases, the SP fracture energy E_{frac} should be calculated as the area under the load-displacement curve up to first load drop. CWA 15627 recommends integrating up to a point where the force has dropped to 80% of its maximum value. However, the determination of SP transition temperature T_{SP} in accordance with CWA 15627 and from energy for the initiation of the first crack was found to be insignificant.

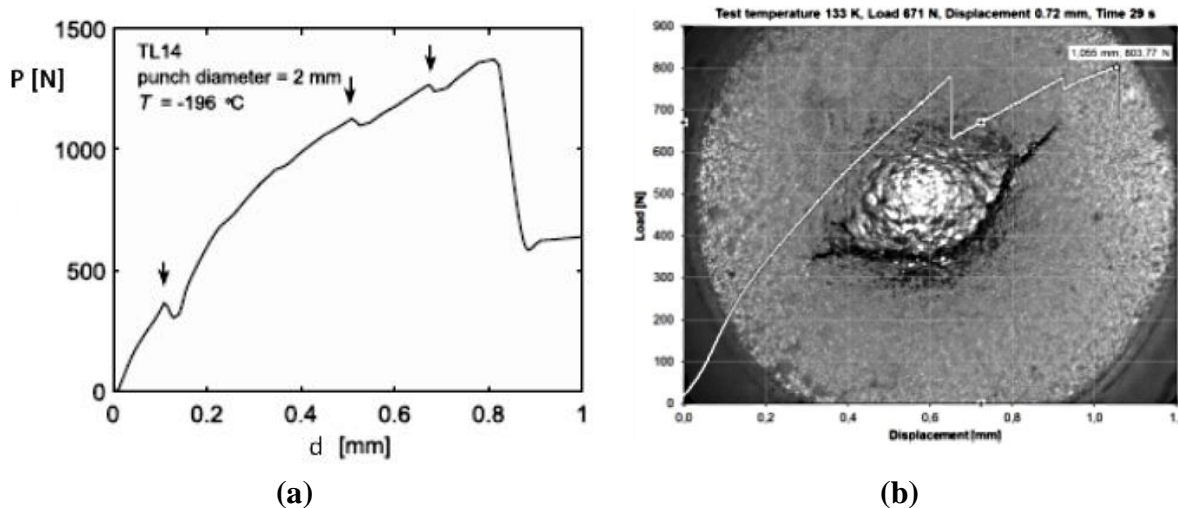


Figure 6: Brittle Material (a) SP force-deflection curve [15]; (b) first circumferential crack [10]

Both the shape of the load – displacement curve and therefore empirical correlations for determination of YS, UTS, DBTT and J_{IC} can be affected first by:

1. Test temperature,
2. Stiffness of the testing machine,
3. Method used for detecting the test specimen displacement,
4. Testing rig (punch tip diameter, dimensions and shape of the chamfer edge, receiving die diameter),
5. Orientation of test disc specimen,
6. Testing material,
7. Initial thickness of the disc test specimen

In Table 2 are shown some advantages/disadvantages of the SPT technique compared to other conventional mechanical tests [11].

Table 2: Advantages of the SPT technique and comparison to conventional mechanical testing techniques [11]

Property Estimation	Advantages of the SPT Technique	Disadvantages/ comparison to conventional mechanical testing
Charpy FATT	Less test material required than CVN specimen test	It is material dependent, but a group of similar materials can be expected to have similar empirical constants (e.g. CrMoV steels)
	Fracture energy is determined during the estimation of FATT	The data can be scattered and empirical constants can be affected by this scatter
	No prior knowledge is required to correlate T _{SP} FATT	T _{SP} can be very challenging to correlate, especially if it is at significantly low temperatures, below -196°C. The specimen is normally cooled using LN ₂

Ferritic steels exhibit a typical sigmoidal curve when impact energy is plotted as a function of test temperature. This sigmoidal curve is used to establish DBTT. SPT results with decreasing temperature also show a ductile to brittle energy transition behavior. DBTT expressed as Fracture Appearance Transition Temperature (FATT) is correlated according to Code with T_{SP} (Small Punch transition temperature), determined from the results of punch tests in the temperature range – 193 °C to +23 °C in the form of equations 2.

$$T_{SP} = \alpha \cdot FATT_{Charpy} \text{ or } FATT_{Charpy} = \alpha \cdot T_{SP} + \beta \quad (2)$$

where α and β are material characteristic constants.

The Small Punch transition temperature T_{SP} is determined according to the Code CWA 15627 as the temperature where E^{SP} has its mean value of the highest and the lowest values in the transition region, by intersecting the smooth curve fitted from the energy versus temperature dependence of fracture energy E^{SP} (Fig.7) [12].

However, numerous studies have shown that the $DBTT_{SP}$ observed from SP testing is significantly lower than the DBTT from Charpy testing. Typical values for α for structural steels have been reported to be around 0.35.

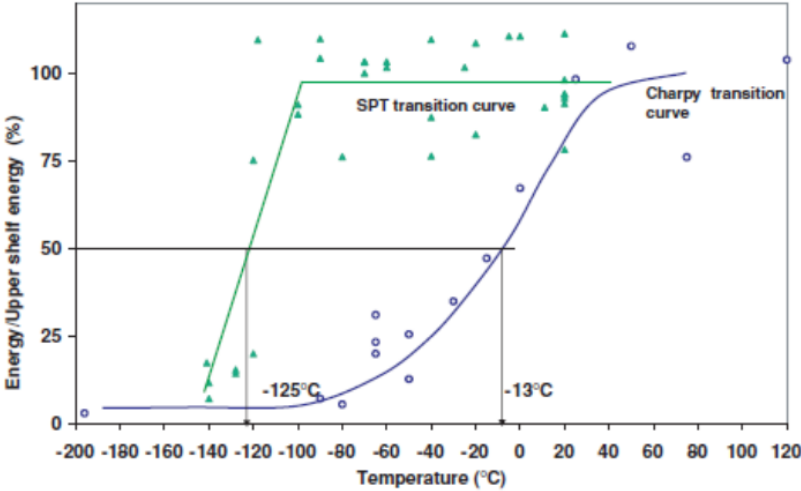


Figure 7: SPT and Charpy Temperature Transition Curves [14]

Another possibility for determining TSP is by means of the fracture strain ϵ_f : (Eq.3) [13]

$$\epsilon_f = \ln (h_0/h_f) \tag{3}$$

where h_f is the specimen thickness after failure adjacent to the area of failure and h_0 is the initial specimen thickness. The transition from ductile to brittle failure is also visible from the change in ϵ_f . The transition temperature determined from ϵ_f is quite similar to that calculated from the energies.

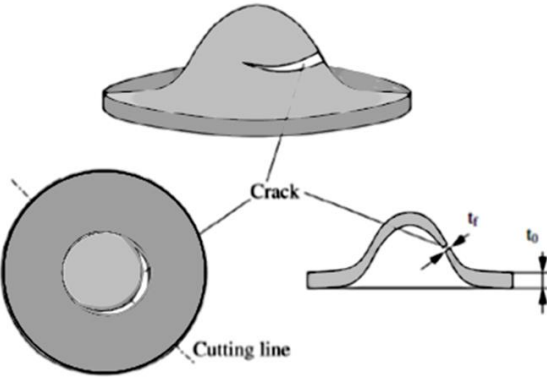


Figure 8: Equivalent Fracture Deformation in a SPT Specimen [6]

To determine the different micromechanisms operating at the different temperatures, a fractographic study of the broken STP specimens was performed [14]. Figure 9(a) gives the fracture surface of the specimen tested at $-85\text{ }^{\circ}\text{C}$, which corresponds to the STP high energy region. This specimen was extensively plastically deformed before failure, and necking is also clearly visible in the hoop direction., near the punch-specimen contact. The fracture pattern is characterized by the presence of microcavities typical of ductile failure (initiation, growth and coalescence of microvoids). Figure 9(b) represents the fracture surface of the SPT specimen tested at $-140\text{ }^{\circ}\text{C}$ (SPT low energy region). The failure micromechanism is 100% cleavage as can be expected in the low temperature brittle region. Figure 9(c) corresponds to the SPT specimen teste at $-118\text{ }^{\circ}\text{C}$ (transition region) where ductile and brittle micromechanisms coexit, as microvoids and cleavage planes are simultaneously observed.

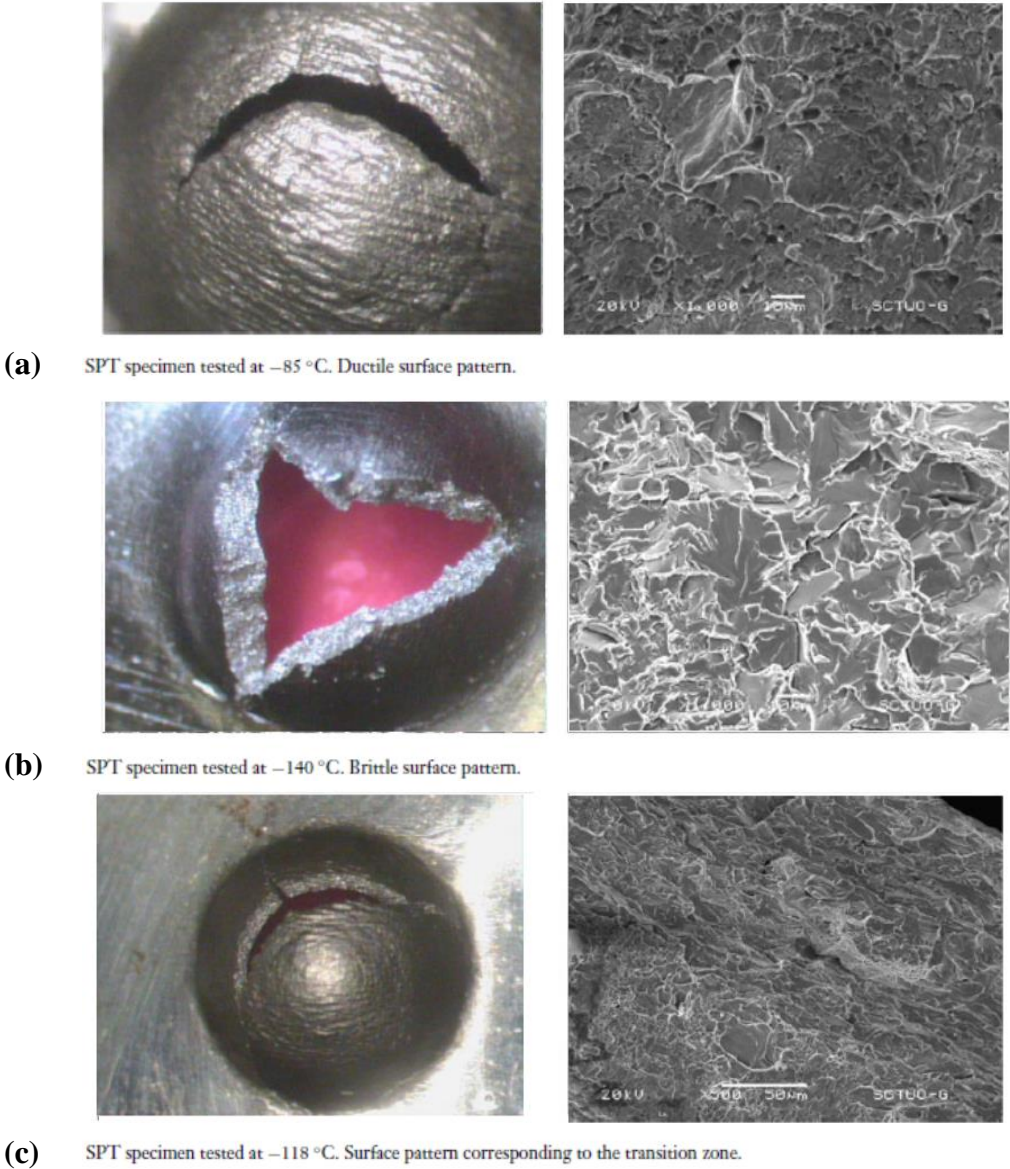


Figure 9: SPT fracture surfaces appearance [14]

To summarize, the transition temperature T_{SP} and thus the empirical correlation FATT vs. T_{SP} can be affected by the following factors:

1. Type of material,
2. Orientation of the disc test specimen,
3. Microstructure inhomogeneity,
4. Method use for the determination of SP fracture energy E^{SP} ,
5. Loading rate,
6. Use of notched disc test specimens (Fig.10) [15, 16].

The main difference between the SP testing technique and standardized impact testing lies in the fact that the SP tests carried out in accordance with the Code use disc-shaped test specimens without a notch. The procedure recommended in the CWA for the determination of T_{SP} in this case, can lead to significant errors in the determination. It is under consideration to include notched disc testing in the proposed standard on SP testing.

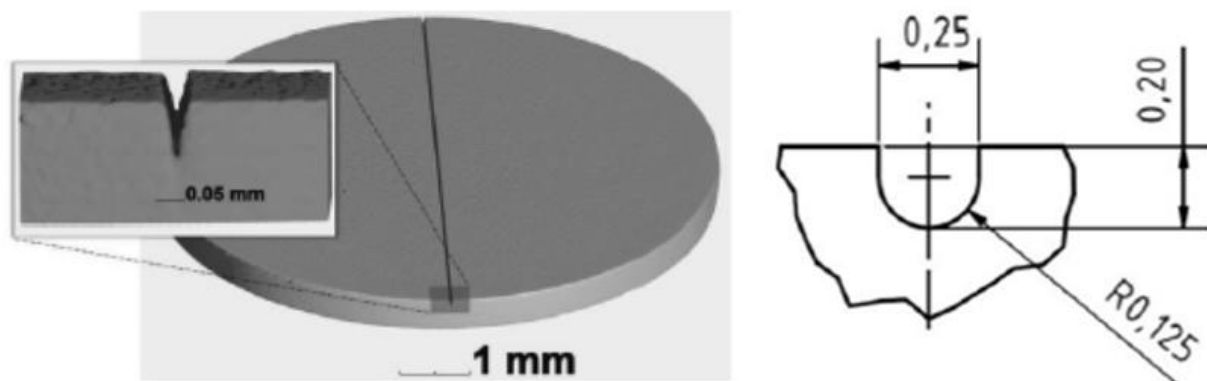


Figure 10: Notched Disc SPT specimen [15, 17]

Although the presence of the notch will undoubtedly play a role in crack initiation results show that the effect of the notch in the Charpy test is much more dominant, indicating that the main effect in the notched SP test lies with the crack propagation. As a result, the transition temperature found in the notched tests are not so much affected by the notch, but the fracture energies are certainly reduced for the notched specimens (Fig.11). This could simply be caused by the difference in ligament length [17].

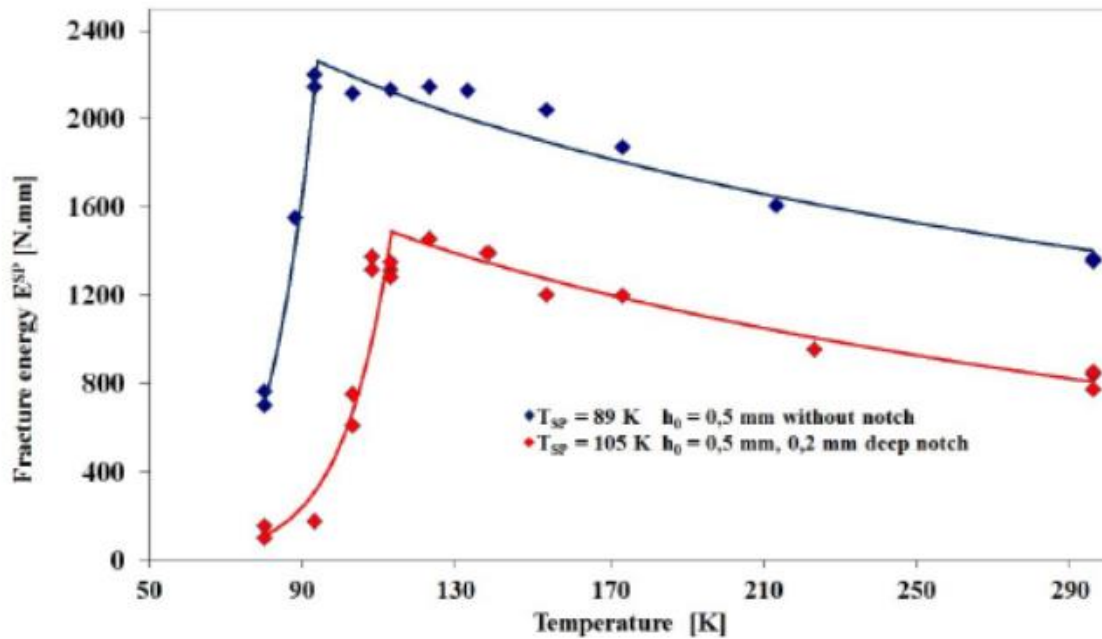


Figure 11: Small Punch Transition Temperature for plane and notched disc specimens [17]

The obtained results confirmed the ones of other works in that the presence of a notch in a SP disc is insufficient to increase the transition temperature significantly and certainly not to the values obtained by Charpy testing. They reveal some evidence that the notch reduces the energy for initiation. Test on a notched disc is more a test of crack growth.

4. THE MASTER CURVE

The Master Curve (MC) is a probabilistic approach that enables the direct estimation of fracture properties in the transition zone of ferritic steels. It enables the characterization of the ductile to brittle transition region (DBTR) with a reduced number of tests, thanks to a combination of mechanistic modelling and a statistical approach (Fig.12) [18].

According to this approach, a mathematical model and a single parameter can define the dependence of fracture toughness with temperature: the reference temperature (T_0). It represents the temperature at which the median of the K_{Jc} distribution (Eq.4) from 1T (25 mm thick) size specimens is equal to $100 \text{ MPa}\cdot\text{m}^{0.5}$ and is the only material dependent parameter required.

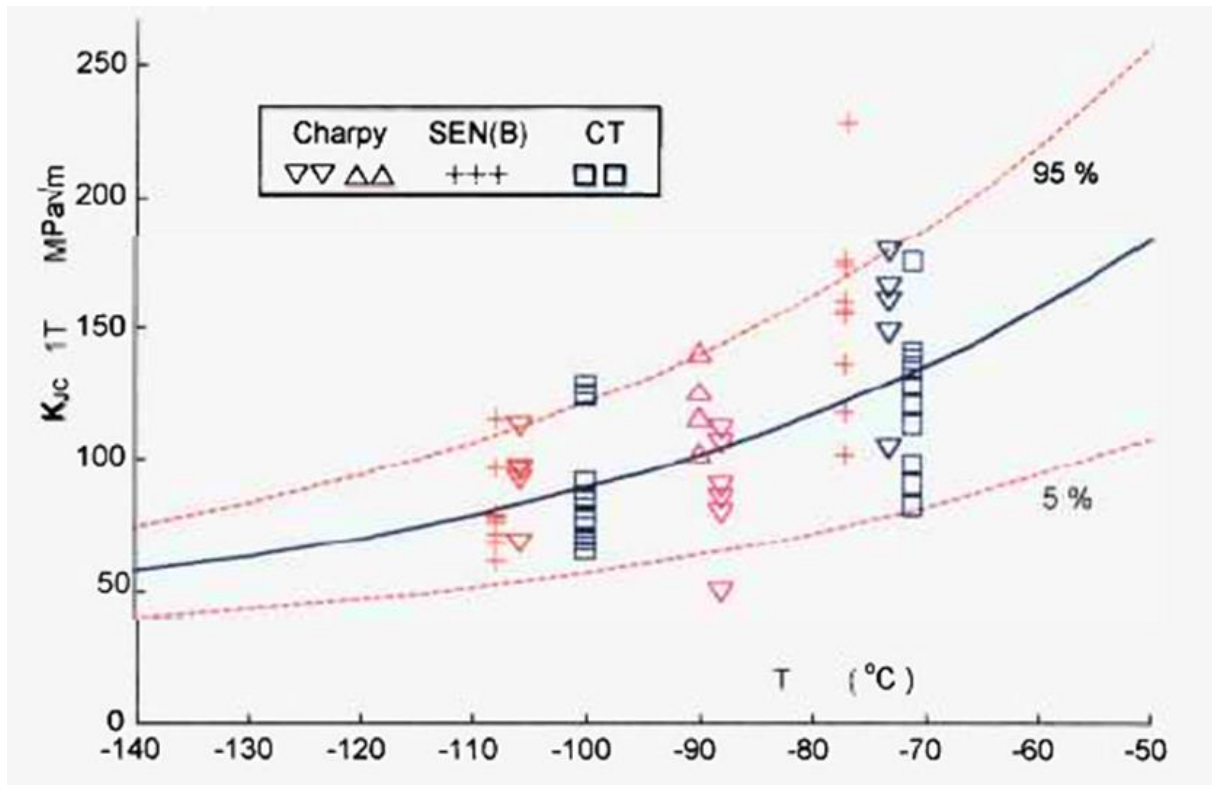


Figure 12: Master Curve and its Associated Limit Curves [18]

$$K_{Jc} = 20 + \left[11 + 77 \cdot e^{[0.019 \cdot (T - T_0)]} \right] \left(\frac{25}{B} \right)^{\frac{1}{4}} \left[\ln \left(\frac{1}{1 - P_f} \right) \right]^{\frac{1}{4}} \quad (4)$$

The determination of T_0 has usually been performed by means of conventional fracture toughness tests (Fig.13). These require relatively large volumes of material, which is often not feasible since the material in the surveillance capsules is becoming scarce. As a result, large efforts are being done to develop small-scale testing techniques in combination with MC, in order to further optimize the material.

Regarding the specimens, the recommended geometry is an 8 mm-diameter discs with 0.5 mm thickness, but 10 x 10 mm square specimens of 0.5 mm-thickness have also been employed (Fig. 14). The use of this last geometry eases the orientation of the specimens and enables the direct reutilization of already tested Charpy specimens. To estimate fracture toughness, some authors have used a modification of the specimens with a lateral notch of circa 4.4 mm and 0.15 mm- radius.

In addition, this approach considers the statistical effects of the thickness of the specimens on the fracture toughness values, as well as the scatter of the results in the DBTR. In the SPT case, considering that the thickness of SP specimens is 0.5 mm, this implies that the K_{Jc} estimation from small punch tests corresponding to a 1T specimen, K_{Jc}^{SP} [1T], is obtained from the K_{Jc}^{SP} corresponding to 0.5 mm-thickness, K_{Jc}^{SP} [0.5], (Eq.5) [19]:

$$K_{Jc}^{SP} [1T] = 20 + [K_{Jc}^{SP} [0.5] - 20]. (0.5 / 25.4)^{1/4} \quad (5)$$

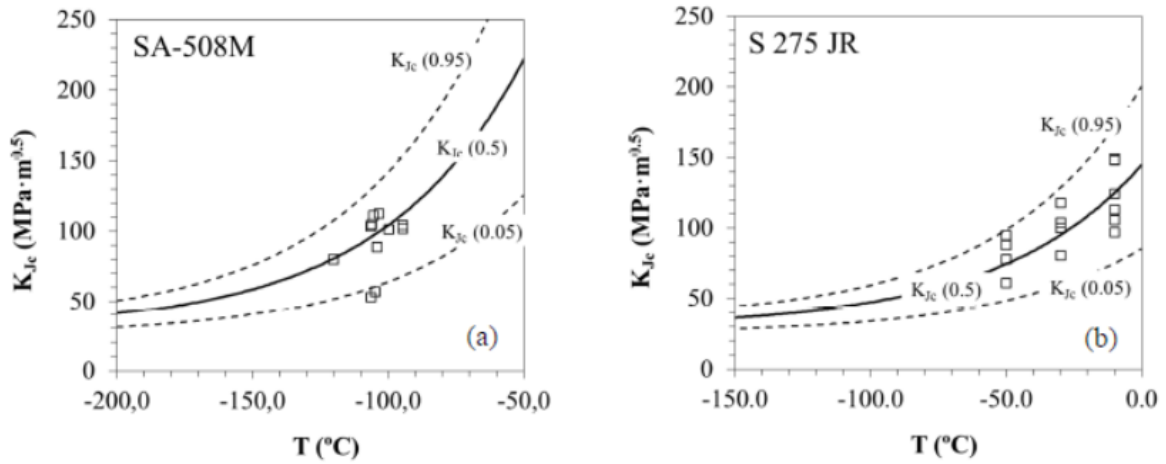


Figure 13: Master Curves using standard specimens [12]

It was observed in the experiments that those specimens suffering a sudden load drop at maximum force exhibit a brittle fracture, while those with a discontinuity on its slope exhibit a mixed mechanism of fracture, with cleavage present on it after some ductile tearing. Finally, tests performed at room temperature which do not show such discontinuity exhibit a fully ductile fracture. Consequently, it is recommended to analyze the micro-mechanisms in case of doubt after the performance of the small punch tests.

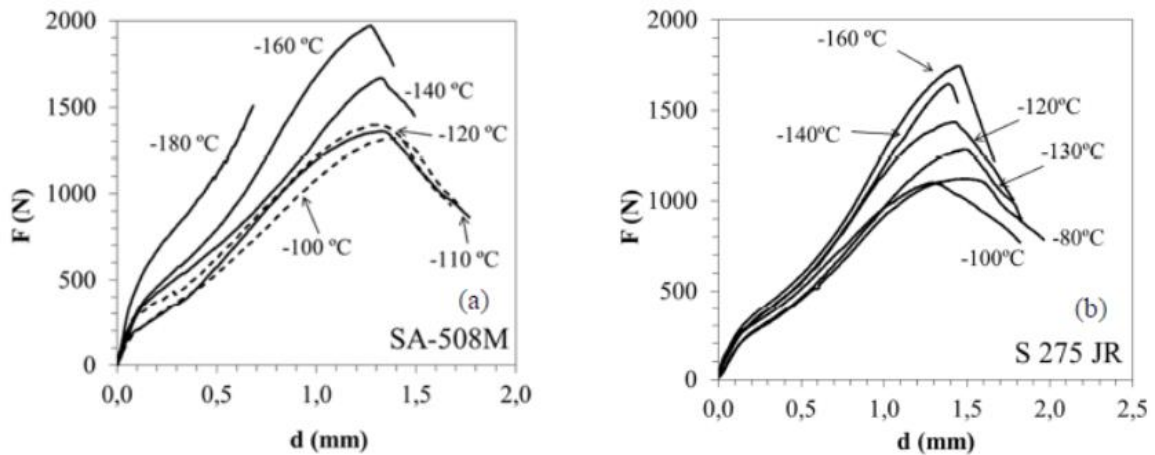


Figure 14: SPT Curves for notched specimens [12]

The reference temperature obtained by means of small punch tests is lower than those values obtained by means of full-scale conventional tests. Consequently, a relationship between both reference temperatures needs to be established. The reference temperature, in K, obtained by means of a small punch tests is approximately half of the corresponding value obtained by means of full-scale conventional tests (Fig. 15). The difference between T_0 and T_0^{SP} might be not related to absolute specimen size, but to the influence of the specimen geometry and loading mode on the cleavage toughness.

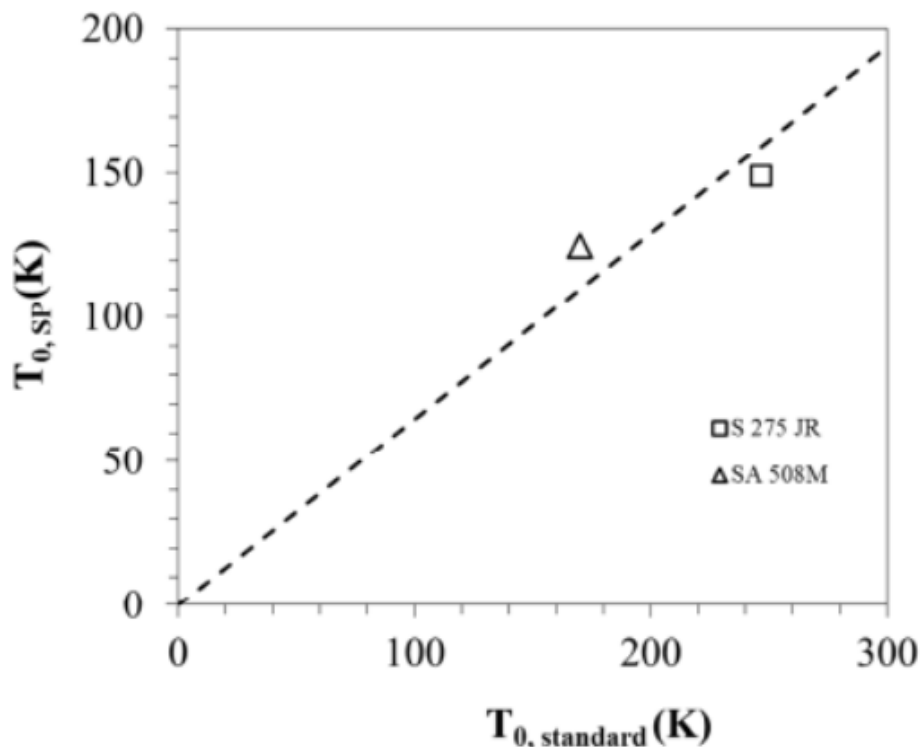


Figure 15: T_0 values comparison between standard and SP specimens [12]

5. CONCLUSIONS

Major benefits of small punch test method are:

1. It can be applied as a virtually non-destructive tool to monitor in-service components in industrial plants.
2. It enables determination of the tensile, fracture, and creep characteristics of materials at critical locations of the components.
3. The test itself is rather simple to perform and almost inexpensive.
4. It gives the possibility to study locations such as interfaces, coatings, welded joints [base metal (BM), heat-affected zone (HAZ), and weld metal (WM)] and exotic materials, e.g. anisotropic.

Of considerable importance in the application of SP fracture testing is the acceptance of plant operators and regulators whether they can rely on the α values obtained in predicting the FATT or DBTT properties for components, in particular where degradation in long term properties may be expected for example through irradiation, creep, or other microstructural damage.

Issues like the reliable transfer of the DBTT determined from SP tests and established standard test still needs further research. Finite element analysis (Fig.16) is expected to be an essential tool for the further development of the technique as it gives insight into the test method itself at a higher level of detail than can be achieved experimentally.

Establishing international standards for SP testing is necessary to ensure comparability of test results between different organizations.

The authors of this study are part of a group of researchers from an ongoing project at Ipen, to investigate the use of miniaturized specimens to assess the structural integrity of nuclear power plant components.

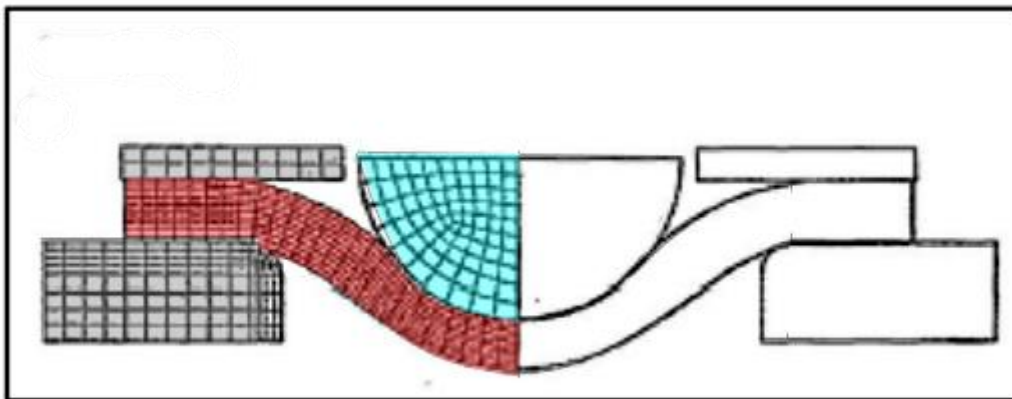


Figure 16: Finite Element modeling of SP Test

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