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# Current status of the small punch test standardization within the ASTM

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**Abstract:** Since 2014, considerable attention has been paid to the standardization of small punch test technique within the American Society of Testing and Materials (ASTM). In 2016 large inter-laboratory study has been launched within the ASTM subcommittee E10.2 - Behavior and Use of Nuclear Structural Materials, involving 12 laboratories and 6 evaluated structural materials from the nuclear power plant components. Paper describes the current status of ASTM standardization, results of the inter-laboratory study, lessons learned and open questions remaining to be solved for the successful completion of the standardization process.

**Keywords:** small punch test; standardization; ASTM

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## 1. Introduction

In the terms of industrial components operational life management, the current trend of lifetime extension requires precise information of structural materials degradation. Conventional methods of mechanical testing are usually based on the use of large specimens and higher consumption of structural materials, whose availability and volume is often limited. For the determination of material properties, sampling of the necessary volume of material is in most cases connected with affecting the integrity or even destruction of the assessed component. Moreover, in the field of nuclear power energy sector, several components are not usually covered by programs of surveillance testing samples, e.g. reactor pressure vessel internals.

Innovative testing method of Small Punch Testing (SPT) is based on the determination of material properties from miniaturized testing specimens and its semi-destructive approach is very promising for the possibility of present data base of materials testing results enlargement. Even though the SPT has been introduced already in 1981 [1], currently the method is unfortunately not supported by the fully-fledged and globally accepted normative document. In 2007, the first initiative towards the preparation of the full standard was established, when the pre-normative document CWA15627 was created as a result of the cooperation within the European community of testing laboratories [2]. At present, the development of European standard continues within the European Committee for Standardization (European Committee for Iron and Steel Standardization - ECISS, subcommittee TC101) with the expected publication of the standard in 2019 [3].

Since 2014, after the 6th International Symposium on Small Specimen Test Techniques (January 2014, Houston, USA), considerable attention has been paid to the standardization of small punch test technique within the American Society of Testing and Materials (ASTM). In 2017, large inter-laboratory study (ILS) was launched to establish the precision statement for the proposal of a new ASTM test. This activity is performed within the Committee E10 on Nuclear Technology and Applications, Subcommittee E10.02 on Behavior and Use of Nuclear Structural Materials. Twelve laboratories around the world participated in this ILS where six different structural materials were tested. Together, more than 460 test specimens were included in this program and more than 3,200 test results were obtained and analyzed.

## 2. Laboratories

For the purpose of inter-laboratory study, group of testing laboratories was created to fulfil the ASTM requirements on the sufficient volume of obtained results. To assure the validity of the round robin testing 12 testing laboratories were involved. In the inter-laboratory study, ÚJV Řež, a. s. is the administrator and supplier of testing materials. Detailed overview on the participants is available in Table 1.

**Table 1.** Testing laboratories involved in the inter-laboratory study.

Laboratory	Country
Helmholtz-Zentrum Dresden-Rossendorf (HZDR)	Germany
VÚJE a.s.	Slovakia
ÚJV Řež, a. s.	Czech Republic
COMTES FHT	
EC JRC Petten	Netherlands
CIEMAT	Spain
Swansea University	Great Britain
National Nuclear Laboratory (NNL)	
Central Research Institute of Electric Power Industry (CRIEPI)	Japan
Korea Atomic Energy Research Institute (KAERI)	Korea
Indira Gandhi Centre for Atomic Research (IGCAR)	India
East China University for Science and Technology (ECUST)	China

### 3. Materials and Methods

For the inter-laboratory study 6 different materials from the nuclear industry were selected (see Table 2). Chosen materials cover wide range of mechanical properties – from basic carbon steels (e.g. 22K) up to highly-alloyed stainless steels (e.g. 10Ch11N20T3R). Also material A533B (JRQ), the IAEA correlation monitoring material [4], was included for its wide use in the programs dedicated to the nuclear materials degradation research. All materials were supplied by the ÚJV Řež, a. s. to the participants in the form of grinded cylinders with the diameter of 8 mm and length approximately of 50 mm to ensure the sufficient volume of material for specimens preparation. From the material A533B (JRQ), ÚJV Řež, a. s. also prepared complete testing samples that were also shipped to participants to determine the influence of the machining and finishing procedures on the test results.

**Table 2.** Chemical composition of materials included in the inter-laboratory study (mass %).

Material	C	Si	Mn	P	S	Cr	Ni	Ti	Mo	Cu	V
<b>A533B (JRQ)</b>	0.18	0.20	1.42	0.020	0.017	0.12	0.84	-	0.51	0.14	0.002
<b>15Kh2MFA</b> <sup>1</sup>	0.16	0.20	0.50	0.020	0.020	2.84	0.11	-	0.70	0.06	0.300
<b>08Kh18N10T</b> <sup>1</sup>	0.06	0.62	1.32	0.022	0.003	17.55	10.80	0.31	0.25	0.08	0.046
<b>22K</b> <sup>1</sup>	0.20	0.23	0.76	0.005	0.001	0.22	0.32	0.01	0.10	0.13	0.020
<b>10Kh11N20T3R</b> <sup>1</sup>	0.03	0.29	0.65	0.007	0.002	10.48	18.40	2.05	0.04	0.04	0.038
<b>COST F</b>	0.12	0.07	0.57	0.010	0.007	10.58	0.37	-	1.51	-	0.190

All participants were given instructions and recommendations how to perform the preparation of testing samples and how to conduct the testing and data acquisition to ensure the comparability of testing outputs. For the testing specimen, geometry of 8 mm diameter and 0.5 mm thickness was selected. Specimens were recommended to be prepared by the electric discharge cutting and subsequent grinding and finishing. Other methods of initial cutting were also allowed, however the removal of material thickness affected by the cutting procedure was necessary to achieve, e.g. EDM cutting of intermediate product with thickness 0.65 mm, grinding on abrasive paper

<sup>1</sup> Equivalent materials - 15Kh2MFA (15CrMoVA), 08Kh18N10T (AISI 321), 22K (ASME SA 515 Grade 70), 10Ch11N20T3R (11Cr-20Ni)

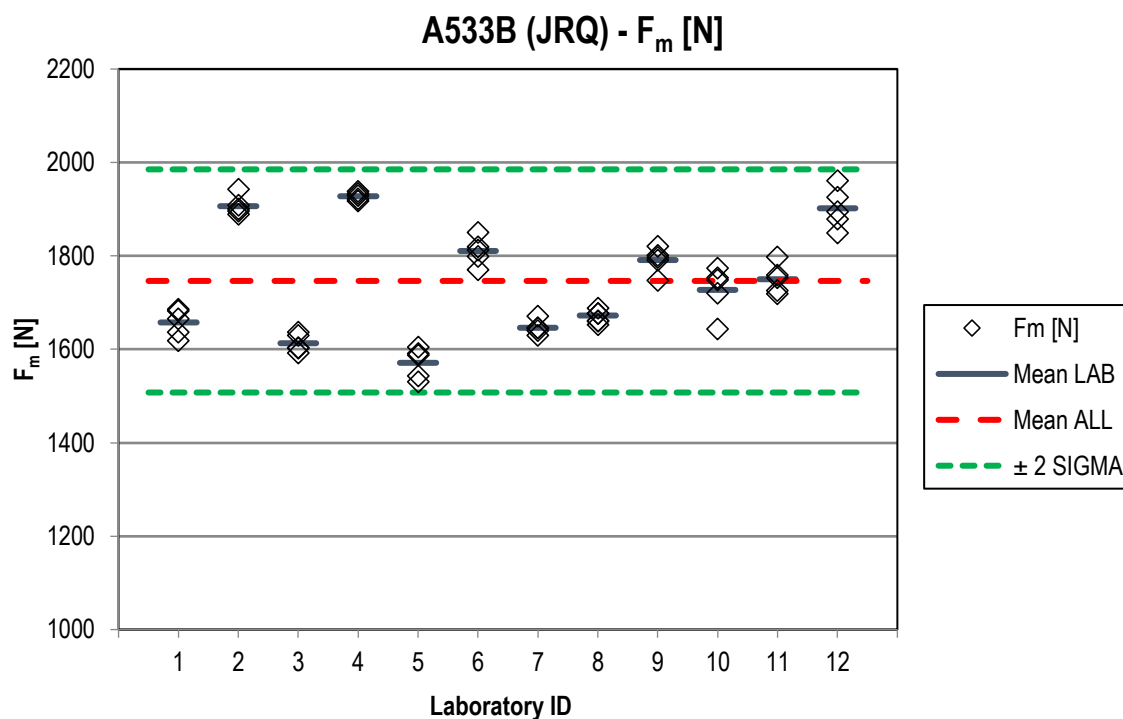
with a recommended abrasive grit size designation P320 followed by fine grinding (P1200) to the final thickness with an accuracy of  $\pm 1\% h_0$ .

**Table 3.** Requested testing data to be recorded for each specimen.

$F_m$ [N]	Maximum force recorded during the SP test
$F_e$ [N]	Force characterizing the transition from linearity to the stage associated with the spread of the yield zone through the specimen thickness (plastic bending stage)
$u_m$ [mm]	Punch displacement corresponding to the maximum force $F_m$
$u_f$ [mm]	Punch displacement corresponding to fracture at 20 % force drop, i.e. $F_f = 0.8 F_m$
$E_{SP}(E_{TO}, E_{PL})$ [J]	SP fracture energy obtained from the area under the force punch displacement curve up to fracture at 20 % force drop ( $F_f = 0.8 F_m$ )
$\epsilon_f$ [-]	Effective fracture strain

#### 4. Evaluation

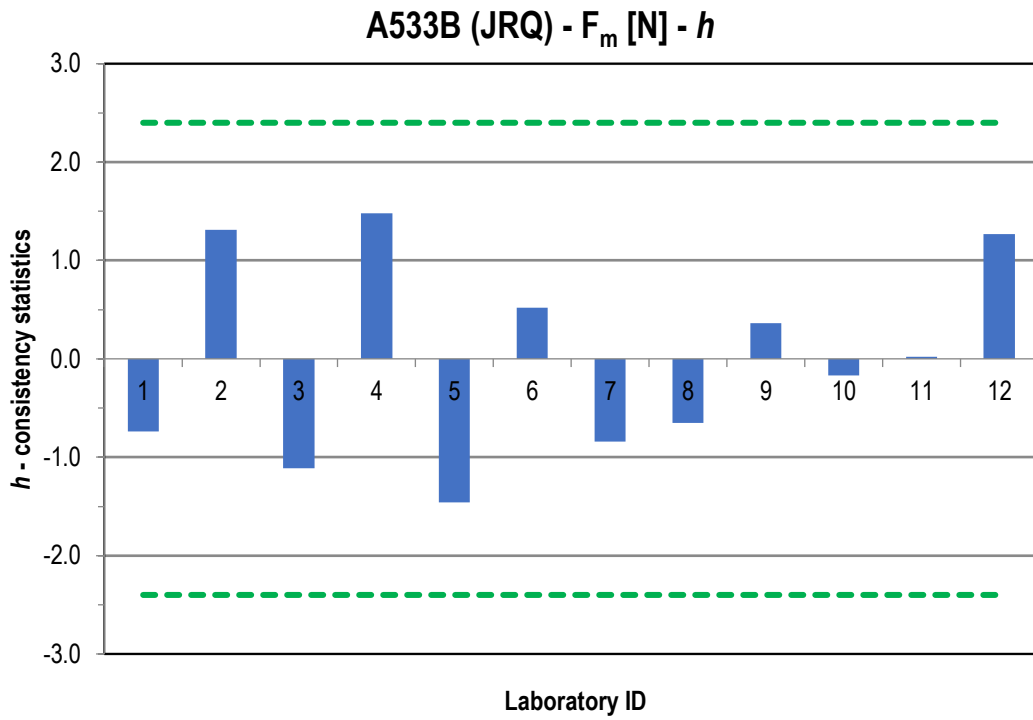
During the 2017, participants of the ILS submitted testing data to the ÚJV Řež, a. s. for further statistical evaluation. The data collection phase of the inter-laboratory study was completed in October 2017. Subsequently, extensive evaluation of the inter-laboratory study was carried out in accordance with ASTM E691-15 for inter-laboratory testing requirements [5], where in addition to a standard statistical evaluation of results the data consistency within a laboratory and between laboratories should also be assessed. Together the total evaluation consisted of 56 excel files and 112 separate charts. Typical example of data evaluation is given on Figure 1 – Figure 3.



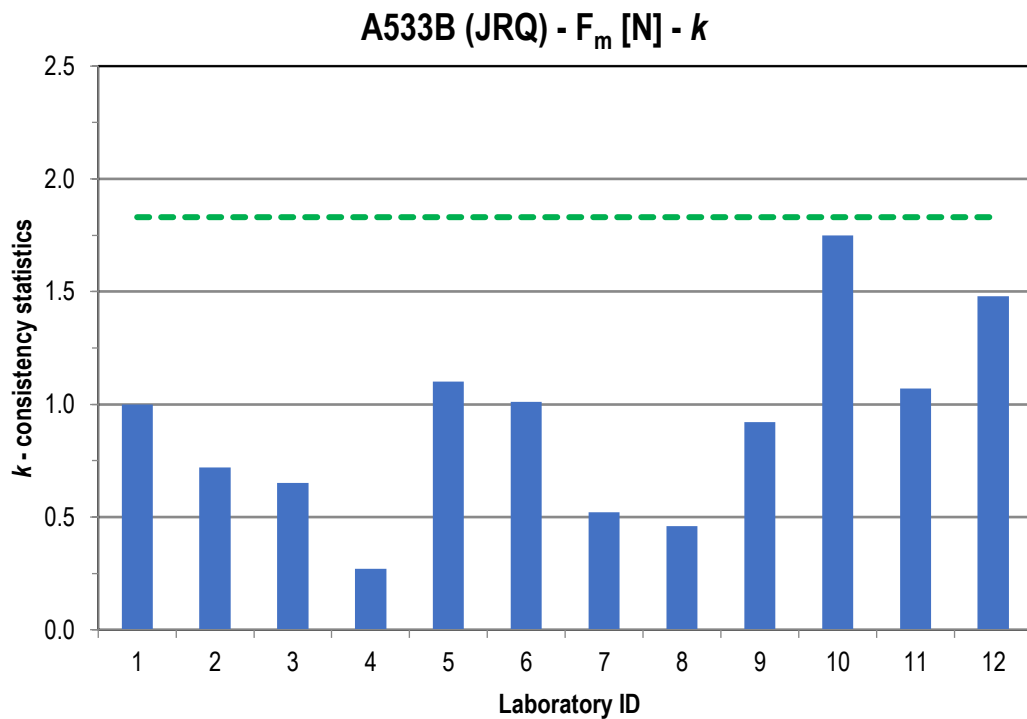
**Figure 1.** Comparison of SP maximum force results between participating laboratories (A533B JRQ).

Also participating laboratories have sent supplementary information related specimen preparation technique, testing procedures and used equipment to enable more detailed comparison and determine possible open issues to be address during the preparation of the final standard. Key areas of the testing procedure with existing open issues to be addressed include – type of used indenter (ball / punch), geometry of the lower die receiving hole edge (radius / chamfer) and the way of the specimen final thickness measurement for the fracture strain determination.

Based on the evaluated inter-laboratory comparison, changes to the draft WK61832 were proposed so that they can be incorporated into the final version of the standard during the balloting process in the period from May 2018.



**Figure 2.** Inter-laboratory consistency statistic  $h$  for maximum force  $F_m$  and A533B JRQ material.



**Figure 3.** Intra-laboratory consistency statistics  $k$  for maximum force  $F_m$  and A533B JRQ material.

## 5. Discussion

The paper summarizes the current status of inter-laboratory study ILS1408 ongoing within the ASTM, committee E10 on Nuclear Technology and Applications. Based on the evaluation participating testing laboratories results the research report was created with the accordance with the ASTM procedures. The precision statement was determined through the statistical examination of 3241 test results from 463 tests from 12 laboratories, on 7 groups of materials. The report was after completion submitted in the January 2018 to the ASTM approval and subsequently subcommittee ballot of the draft standard was prepared. Successful balloting process will address current uncertainties in the testing procedure and will be a basis for the preparation of fully-fledged ASTM standard.

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