APMP.M.MM-S1

Supplementary Comparison on Tensile Properties Measurement

Final report for APMP.M.MM-S1

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Abstract

This report describes the results of the supplementary comparison, APMP.M.MM-S1, between 4 participants. KRISS was the pilot laboratory of this comparison. The reference material with tensile strength of 636 MPa was prepared by the pilot laboratory, KRISS (Korea) and provided to each participating lab.

The consistency of this measurement comparison was checked, and, based on the consistency examination of the measurement, a 600 MPa level tensile strength standard was defined.

1. Introduction

This report describes an APMP (Asian Pacific Metrology Program) supplementary comparison (SC) of tensile strength measurement. The comparison is designated as APMP Project and recognized as APMP.M.MM-S1 in the key comparison database (KCDB). The comparison comprised four participants including the pilot laboratory (see Table1).

This comparison was proposed in 2011 by the WGMM (Working group on Material Metrology) at the annual meeting of APMP, held in Kobe, Japan. The supplementary comparison commenced in 2012 with five participants, however only four participants provided measurement results. Table 1 lists the comparison participants.

KRISS was the pilot laboratory, and produced and provided the Reference Material (RM), which was developed in KRISS, with reference tensile strength of 600 MPa level.

In this comparison, tensile strength measurement results determined at each participant are reported, and a reference value is defined through the consistency check of the measurement results. The elastic modulus and yield strength properties could be measured along with this tensile strength measurement in this comparison, but, in this report, only the tensile strength measurement results are compared.

Table 1 List of participants in the comparison

Laboratory		**Economy**
Korea Research Institute of Standards and Science	KRISS	Republic of Korea
Federal Institute for Materials Research and Testing	BAM	Germany
National Institute of Standards and Technology	NIST[1)]	USA
Kenya Bureau of Standards	KEBS[2)]	Kenya
National Institute Metrology of Thailand	NIMT[3)]	Thailand

NOTE) 1) started participating in the comparison in 2013; 2) started participating in the comparison in 2014; 3) tried to participate in this comparison two times, in 2012 and 2016, but did not submit a final results due to difficulties in establishment of a standard testing system

2. Description of tensile strength measurement and reference material

2.1 Definition of the tensile strength to be measured

Tensile strength, R_m, at room temperature is defined as the stress value determined at peak load, P_m, obtained from the specimen pulled with a tensile force at a predefined tensile strain rate, as shown in Fig.1. The tensile strength can be presented as the following;

$$R_m = P_m / A \tag{1}$$

Here, A is a cross-sectional area at the testing section of the tensile specimen.

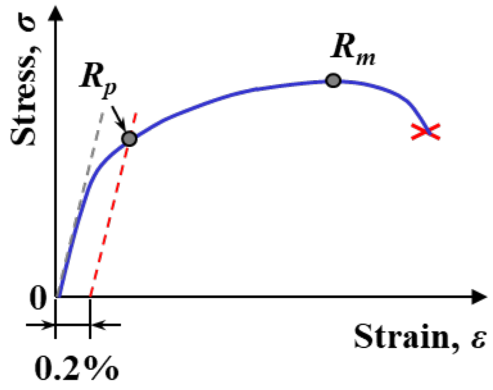

Fig. 1 Stress-strain curve obtained from the tensile strength measurement.
(Here, R_p and R_m represent 0.2% offset proof strength and tensile strength, respectively.)

2.2 Measurement Procedure

The protocol for the comparison is provided in Appendix A. This measurement was recommended to be carried out according to ISO 6892:2009 Metallic Materials - Tensile Testing at Ambient Temperature[1].

Specially, the testing speed and control mode for the tensile loading was recommended to be as follows;

- Up to 2 % tensile strain, the measurement is recommended to be performed at a strain rate of 0.000 25 s^{-1} ± 20 %,
- Above 2 % tensile strain, the measurement shall be kept at a constant crosshead separation rate of 0.4 mms^{-1} ± 10 % without any interruptions or holds as possible.

2.3 Reference Material provided by pilot laboratory

Reference material used in this comparison was prepared in a single batch and provided by the pilot laboratory, KRISS (Korea), to each participant. Each participant laboratory received 5 reference materials which were labelled and separately packed

[1] This standard was revised to ISO 6892-1:2016[1]

in vacuum, were delivered to each participating laboratory. Fig. 2 represents a photograph of a reference material delivered to the participants. The dimensions of the material are listed in Table 3. .

The properties of the reference material which were determined in the pilot laboratory are showed in Table 3.

Fig. 2 Photograph of a reference material provided to each of the laboratories for the comparison

Table 2 Geometrical details of the reference material

Items	Dimension (mm)
G - Gage Length	50
D - Diameter at Reduced Section	10
R - Radius of Fillet	15
A - Length of Reduced Section	60
L - Overall Length	178
B - Length of End Section	50
C - Diameter of End Section	16

Table 3. Properties of the 600 MPa- level reference material

	Elastic Modulus, E (GPa)	**0.2% Proof strength, R_p (MPa)**	**Tensile Strength, R_m (MPa)**
Reference value	198.09	300.87	636.20
Combined uncertainty	1.89	1.97	0.84

3. Summary of results reported by the participants

3.1 Measurement parameters established by each participant laboratory

In order to obtain precise and accurate tensile properties from the tensile test, various parameters required for the measurement need to be considered. Table 4 shows the parameters and status of the parameters established by each participant laboratory, which were investigated according to the questionnaire prepared by the pilot lab. (see Appendix B) All participants prepared the measurement systems and instruments calibrated according to the national standard established in the respective nations or the international standard relevant to each measurement quantity. For most participating labs, wedge grips were used for holding the specimen, while, in KRISS, collet grips were equipped. The respective participant labs were required to estimate the load measurement uncertainties according to ISO 6892:2009 Metallic Materials - Tensile Testing at Ambient Temperature[1].

Table 4 Status of measurement parameters established by the participants

Items	KRISS	BAM	NIST	KEBS
Possible Test Control Methods	C , E	C , E	C , E	C , E
Alignment measurement method	I	I	I	N/A
Measurement of Stiffness of loading train in Testing Machine	A	N/A	N/A	N/A
Gripping Type	C, W	W	W	W
Measurement uncertainty of load	A	A	A	A
Measurement uncertainty of strain	A	A	A	A
Calibration of Load Cell	A	A	A	A
Calibration of Extensometer	A	A	A	-
Load capacity of the machine (kN)	100	100	100	-
Range of Extensometer (working range/gage length) (mm)	5/50	5/50		
Data Handling Method	M, C	M	M, C	M, C

NOTE) Control Method: C: Cross-head (displacement) control, E : extensometer (strain) control

Alignment measurement: I : Instrumented cell, N/A : Non-available

Gripping type : W : wedge grip, C : Collet grip

Data Handling method: M: Manual data treatment C: Computerized Data Management system (including commercial software provided by machine manufacturer)

Etc. : A: Available

3.2 Tensile strength determined from the participants

Each participating laboratory measured the tensile strength of 5 samples provided by the pilot laboratory, KRISS. The average and standard deviation of the strength values obtained at the respective participants were calculated and are summarized as shown in Table 5.

Average values of the tensile strength measured from KRISS and BAM were identical, while the values from NIST and KEBS were relatively greater than those from KRISS and BAM. Standard deviation of the strength measured from KRISS and BAM showed to be less than 1, while those from NIST and KEBS were greater than 1. Those values were plotted in Fig. 3.

Table 5. Average, standard deviation and uncertainties of the strength values reported by each participant

Participant	Tensile Strength, R_m			
	Average	s[1]	u_c[2]	u_c[3]
KRISS	635.86	0.23	0.40	0.51
BAM	637.94	0.73	0.64	1.05
NIST	644.08	4.02	-	4.62
KEBS	640.70	1.16	0.47	1.67

NOTE) [1] S: standard deviation

[2] u_c : combined uncertainty provided by each organization

[3] u_c : combined uncertainty considering uncertainty due to measurement repetition

$$u_{c,i} = \sqrt{u_i^2 + \frac{(t_p \cdot s)^2}{n}}$$

Here, t_p represents the value of the Student's t-distribution for a confidence level p and n number of repetition.

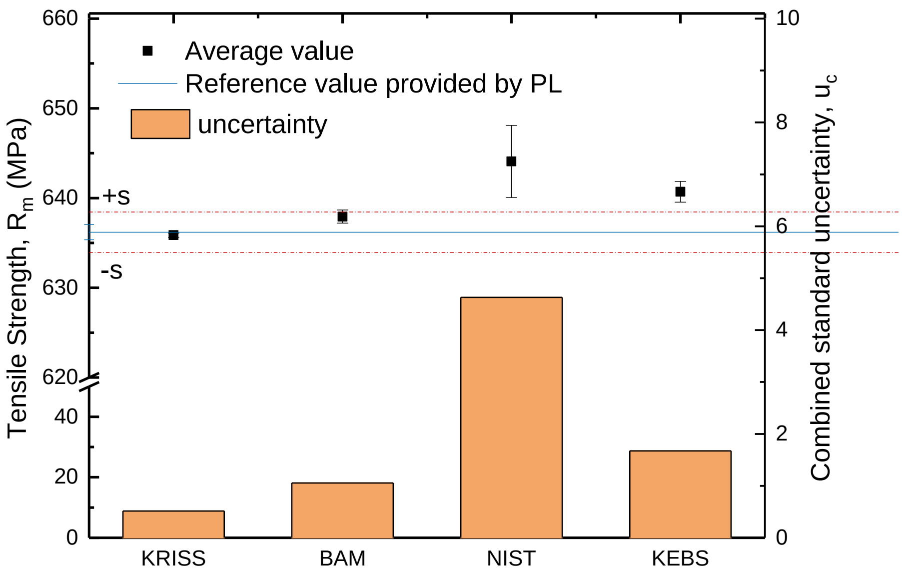

Fig. 3. Tensile strength along with standard deviation and combined standard uncertainty from each participant.

The strength values measured at each participant were compared to the reference value as the difference ($R_{m,avg}$ -$R_{m,0}$) between the reported tensile strength value ($R_{m,avg}$) and the reference strength value ($R_{m,0}$). Here, the reference tensile strength is 636.20 MPa (SD=2.25 MPa), as listed in Table 2. The comparison is presented in the Table 6, and plotted in Fig. 4.

Table 6. Difference between the reported tensile strength value ($R_{m,avg}$) and the reference strength value ($R_{m,0}$)

Participant	**$R_{m,avg}$-$R_{m,0}$ (MPa)**
KRISS	-0.33
BAM	1.74
NIST	7.89
KEBS	4.50

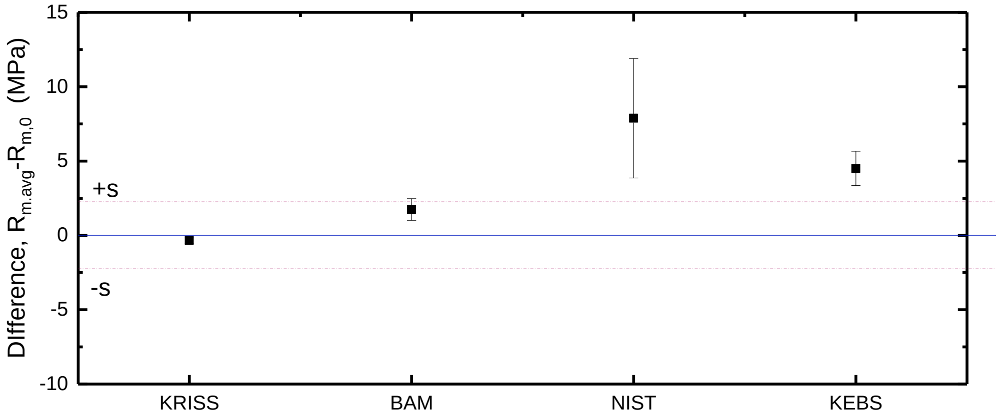

Fig. 4. Difference ($R_{m,avg}$ -$R_{m,0}$) between the reported strength value ($R_{m,avg}$) and the reference strength value ($R_{m,0}$)

4. Determination of reference value

The reference value of the tensile strength for the reference material provided from the pilot laboratory can be redefined from this comparison results. The value, $R_{m,r}$, can be taken to be a weighted mean of the measured strength differences of each participant. The squared standard uncertainties reported by the participants were used as weights. The standard deviation of the weighted mean could be calculated using the uncertainties of the participating laboratories according to the following equations.

$$R_{m,r} = \frac{\sum R_{m,i}/u^2(R_{m,r})}{\sum 1/u^2(x_i)} \tag{2}$$

$$\frac{1}{u^2(R_{m,r})} = \sum \frac{1}{u^2(R_{m,i})} \tag{3}$$

The weighted mean and the associated uncertainty of the all measurements determined using the Eq. (2) and (3) were 636.64 MPa and 0.44 MPa, respectively.

According to Procedure A, the consistency of this measurement result comparison was checked by applying a chi-squared test. Table 6 represents the consistency check of this comparison.

Table 7. Degree of Equivalence and E_n values for all measurements

Participant	DoE		E_n-value [3]	Contribution $\boldsymbol{e_i(R_{m,i})}$ to SS function [4]
	Difference, d_i [1]	Standard uncertainty, $u^2(d_i)$ [2]		
KRISS	-0.78	0.07	1.50	2.31
BAM	1.29	0.92	0.68	1.51
NIST	7.44	21.24	0.81	2.58
KEBS	4.06	2.60	1.26	5.88

NOTE) 1) Difference, $d_i = R_{m,i} - R_{m,r}$,

2) Uncertainty $u^2(d_i) = u^2(R_{m,i}) - u^2(R_{m,r})$

3) Normalized deviation, $E_n = \frac{R_{m,i}-R_{m,r}}{[u^2(R_{m,i})-u^2(R_{m,r})]^{1/2}}$

4) contribution to SS(sum of square) function,

$F_N(R_m) = \sum_{i=1}^{N} e_i(R_{m,i})$, $e_i(R_{m,i}) = (\frac{R_{m,i}-R_{m,r}}{u(R_{m,i})})^2$,

As found from Table 7, E_n values for the measurement results obtained from all NMIs are such that E_n <2. However, the SS function is 12.29 which is greater than the 100α percentage point of the chi-squared distribution. $\chi^2_{N-1,\alpha}$. Here, $\chi^2_{N-1,\alpha}$ for N=4, α =0.05 is 7.82. So, the weighted mean value for the full data set including all NMIs measurements is not acceptable because it does not satisfy the consistency test.

For the largest consistent subset (LCS), one measurement determined from KEBS was excluded. The weighted mean and the associated uncertainty of the all measurements determined using the Eq. (2) and (3) were 636.34 MPa and 0.46 MPa, respectively.

DoE and En values for the data subset can be calculated as presented in Table 8. The En values for the subset measurements are less than 2. The SS function, which is determined as 5.97, also is less than the 100α percentage point of the chi-squared distribution. $\chi^2_{N-1,\alpha}$. Here, $\chi^2_{N-1,\alpha}$ for N=3, α =0.05 is 5.99. Thus, the weighted mean value for the data subset excluding one sub-measurement from the KEBS satisfies the consistency test, and the result is acceptable.

Table 8. Degree of Equivalence and E_n values for the data subset excluding one measurement

Participant	DoE		E_n-value	Contribution $e_i(R_{m,i})$ to SS function
	Difference, d_i	Standard uncertainty, $u^2(d_i)$		
KRISS	-0.48	0.05	1.035	0.86
BAM	1.60	0.90	0.84	2.30
NIST	7.75	21.15	0.84	2.81
KEBS	4.36	3.01	1.26	6.80*

NOTE) * : determined from the data subset excluding data from KEBS

The reference value of the tensile strength was determined as the following;

$$R_{m,r} = 636.34 \text{ MPa}$$

with the corresponding uncertainty, $u(R_{m,r})$ =0.46 MPa. Here, the expanded uncertainty with 95% coverage probability, k=2, is 0.92 MPa.

The differences, $R_{m,avg}$ - $R_{m,r}$ between the reference value and values of the strength from each participant, are represented in Fig. 5.

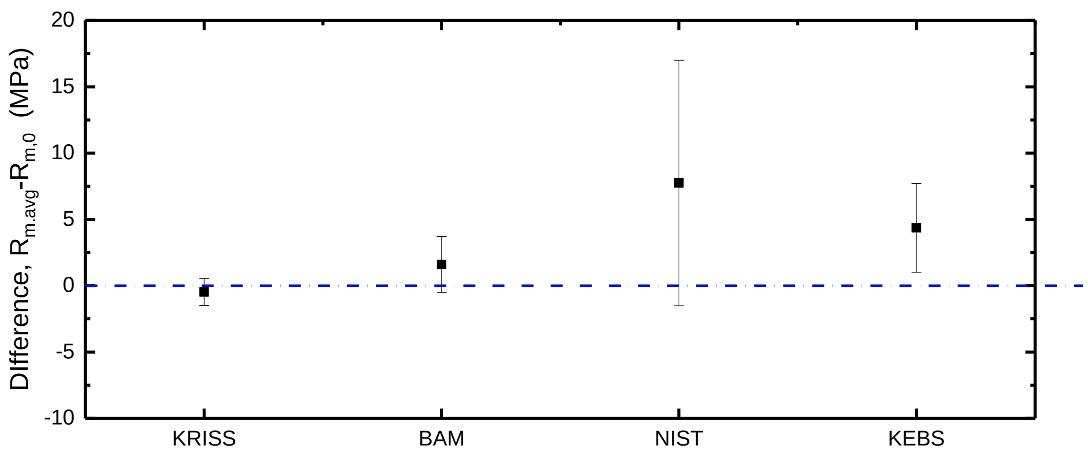

Fig. 5. Difference, $R_{m,avg}$ -$R_{m,r}$ between the reported strength value ($R_{m,avg}$) and the reference strength value ($R_{m,r}$). Here error bar represents the expanded uncertainty of the respective measurements.

5. Summary and Conclusion

Tensile strength measurement was carried out using the reference material provided by the pilot laboratory, KRISS. This comparison started with participation of 5 NMIs, including KRISS, BAM, NIST, KEBS and NIMT, but final measurements participated with 4 NMIs except NIMT were carried out and the results were compared. Consistency of the tensile strength measurement comparison was examined. Measurements from from KRISS, BAM and NIST was found to be consistent. From the consistency examination of the tensile strength measurements, a reference value of tensile strength of 600 MPa class could be defined. The reference value was 636.34 MPa with the expanded standard uncertainty of 0.92 MPa (k=2. 95%).

6. References

[1] ISO 6892-1:2016 Metallic Materials – Part 1: Method of test at Ambient Temperature

[2] Cox M. G. 2002 The evaluation of key comparison data, Metrologia 39 589–95

Appendix A Guideline for Tensile Properties Measurement

(APMP.M.MM-S1: Comparison of Tensile Strength Measurement)

This guideline provides a technical protocol for measuring the tensile properties to all participants in this APMP.M.MM-S-1 comparison.

A.1. Measurement Procedure

The tensile properties shall be measured according to the following procedure;

- Basically, the measurement shall be carried out according to ISO 6892:2009 Metallic Materials - Tensile Testing at Ambient Temperature.
- Specially, the testing speed and control mode for tensile loading shall be specified as follows;
  - In the range from start of the measurement upto 2% tensile strain, the measurement is recommended to be performed at a strain rate of 0.000 25 s-1 ± 20 %,
  - In the range over 2% tensile strain, the measurement shall be kept at a constant crosshead separation rate of 0.4 mm s-1 ± 10 % without any interruptions or holds as possible.

A.2. Sample Code and Specimen Identification

(1) Sample Code

- Material for measurement will be supplied by the pilot lab and shall be used in measurement according to the specified procedure.

(2) Sample

- One sample contains 5 specimens. All of the participants will receive 1 box with 5 specimens.
- Please report test result of 5 specimens

(3) Specimen Identification

Each specimen has its own identification and it is engraved on the surface of the specimen. Be careful during the measurement so that it would not disappear.

(4) If you can not carry out the measurement due to mismatching of your testing machine with the supplied specimens, report the details.

A.3. Elastic Modulus

Please measure and calculate the elastic modulus obtained from the slope of the stress strain curve at the beginning.

A.4. Yield Strength

Even if you observe yield point phenomenon at the stress strain curve, obtain 0.2% offset yield strength (0.2% proof strength, non-proportional extension) and report the result.

A.5. Tensile Strength

Please measure the tensile strength at the maximum load (or stress) at the engineering stress-strain curve and report the result.

A.6. Elongation after fracture and reduction of area

If you can measure the elongation after fracture and reduction of area, please measure these properties and report the results. (These are optional)

A.7. Estimation of measurement uncertainty

1) Please estimate the measurement of uncertainty on the measured values according to the procedure the participants have established in their own way. If each participant does not have, please refer to ISO 6892 or

2) After collecting all data, the procedure will be discussed for estimation harmonization.

A.8. Raw Data

If you can obtain raw data about force, crosshead separation (or displacement), stress, and strain from start of the test upto termination of the measurement, please acquire the data at a sampling rate of no less than 10 samples/s and report it in excel file. If it is not possible, send the stress-strain curve and force-crosshead separation displacement curve).

Appendix B Questionnaire for a APMP SC (supplementary Comparison) on Tensile Properties Measurement

Introduction

This comparison is organised for the purpose of measuring tensile properties- elastic modulus, yield strength and tensile strength. The reference materials will be provided and the tensile properties shall be measured at ambient temperature according to the protocol prepared.

Air temperature: (22 ± 3)°C;
Ambient relative humidity above 50 %;

The detailed measurement protocol will be provided.

Description of the measurement and reference materials:

In this comparison, elastic modulus, yield strength and tensile strength which can be defined as in Fig. 1 will be measured.
The reference tensile specimens which will be provided to each participant will be prepared by the KRISS and the dimension of the specimen is as the followings: diameter at reduced section = 10 mm, gage length = 50 mm, length of reduced section = 60 mm, overall length = 178 mm and diameter of end section = 16 mm.
The load capacity of the testing system is recommended to be above 100 kN

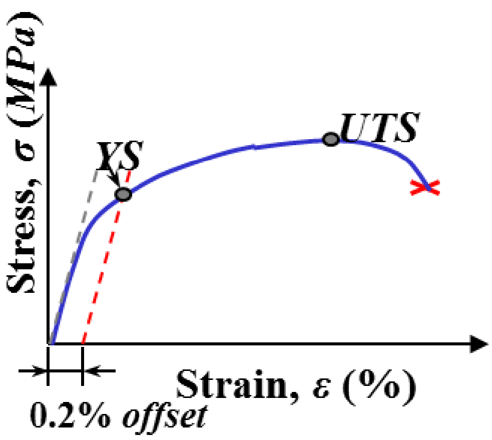

Fig. 1: Definition of tensile properties

Measurement procedure

Basically, the measurement shall be carried out according to ISO 6892:1998 Metallic Materials - Tensile Testing at Ambient Temperature. Specially, the testing speed and c ontrol mode for tensile loading shall be specified as follows;

- In the range from start of the measurement upto 2% tensile strain, the measure ment is recommended to be performed at a strain rate of 0.000 25 s^{-1} ± 20 %,
- In the range over 2% tensile strain, the measurement shall be kept at a constant crosshead separation rate of 0.4 mm s^{-1} ± 10 % without any interruptions or hol ds as possible.

References

1. ISO 6892:2009 Metallic Materials – Tensile Testing at Ambient Temperature.
2. ISO 9513 Metallic Materials – Calibration of extensometer systems used in uniaxial testing
3. BIPM, IEC, IFCC, ISO, IUPAC, IUPAP, OIML; *Guide to the expression of uncertainty in measurement (GUM),* Geneva, 1995.

Feed back Questionnaire

1. Contact Person

(1) Name: ____

(2) Title: ____

(3) Organization: ____

(4) Address: ____

____

(5) Country: ________________________ Zip Code: ______________

(6) Phone: _____________________ Fax: _____________________

(7) E-mail: ____

2. Testing machine

(1) Specification of Testing Machine

- Manufacturer : ____________
- Model : ____________
- Force Capacity: ____________

(2) Force Application System

☐ Electromechanical(or Screw-Driven) Testing Machine

☐ Servohydraulic Testing Machine

☐ Electromechanical Actuator(or Ball-Screw Actuator) Testing Machine

☐ Other (Please Describe: ____)

(3) Possible Test Control Methods (Please check all applicable)

☐ Crosshead or Stroke Control (Constant Grip-Separation Rate)

☐ Stress or Load Control (Constant Stress Rate)

☐ Extensometer Control (Constant Strain Rate)

(4) Alignment

☐ Instrumented cell

☐ Alignment bar

☐ Others or None (if others, please specify: ______________________________)

(5) Stiffness of Loading Train of Testing Machine

☐ Specified, ______________________________)

☐ None

3. Extensometer

☐ Available
- Manufacturer : ____________
- Model : ____________
- Gage Length: ___________ mm
- Maximum Travel Length: ___________ mm

☐ Strain Gage Type

☐ LVDT Type

☐ Optical Type

☐ Other (Please describe: ______________________________________)

☐ Not Available

4. Gripping (Please check all applicable)

☐ Wedge Grips
☐ Collet Grips
☐ Grips for Threaded End Specimens
☐ Grips for Shouldered End Specimens

5. Data Handling

- **Method Used for Determination of Tensile Properties**

☐ Manual Determination from Load-Elongation or Stress-Strain Curves
☐ Computer-Assisted or Entirely Automated Determination
☐ Other (Please describe: ____)

- Extraction of Raw Data on stress-strain and load-extension

☐ Available

☐ None

6. Uncertainty

☐ Available

- Uncertainty in Load Measurement ________________
- Uncertainty in Strain Measurement ________________
- Uncertainty in Tensile properties Measurement

☐ None

7. Additional Comments

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End of Questionnaire