NIST Technical Note XXXX

Repeatability and reproducibility of compression strength measurements conducted according to ASTM E9

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National Institute of Standards and Technology • U.S. Department of Commerce

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Abstract

Ten commercial laboratories participated in an interlaboratory study to establish the repeatability and reproducibility of compression strength tests conducted according to ASTM International Standart Test Method E9. The test employed a cylindrical aluminum AA2024-T351 test specimen. Participants measured elastic modulus and 0.2 % offset yield strength, *YS*(0.2 % offset), using an extensometer attached to the specimen. The repeatability and reproducibility of the yield strength measurement, expressed as coefficient of variations werecv_r= 0.011 and cv_R= 0.020 The reproducibility of the test across the laboratories was among the best that has been reported for uniaxial tests. The reported data indicated that using diametrally opposed extensometers, instead of a single extensometer doubled the precision of the test method. Laboratories that did not lubricate the ends of the specimen measured yield stresses and elastic moduli that were smaller than those measured in laboratories that lubricated the specimen ends. The modulus measured from stress-strain data were reanalyzed using a technique that finds the optimal fit range, and applies several quality checks to the data. The error in modulus measurements from stress-strain curves generally increased as the fit range decreaesed to less than 40 % of the stress range.

Keywords: compression, testing, ILS, ASTM, E9, yield strength, elastic modulus

1 Introduction

Compression testing is a conceptually simple method to establish the uniaxial stress-strain and mechanical behavior of materials. Because the test specimens can be right circular cylinders, they are easy to fabricate. The short gauge length of the test specimen sometimes makes it the only possible geometry for establishing uniaxial properties, for example normal to the plane of a plate. Compression tests can also be used to establish the strength of brittle materials [1, 2] that would be difficult to grip in tension. Kuhn [3] and Chait [4] have reviewed the methods for compression testing, and have demonstrated that although the test is conceptually simple, the user must overcome many experimental difficulties to translate the measured load and displacement curves into accurate stress-strain behavior.

Much of the literature, see Table 1, on implementing the compression test focuses on tests to large strains [7, 8, 12], to establish work hardening behavior beyond the strains at which tension test specimens neck. Other studies are devoted to modelling processes such as upsetting [5, 14, 18, 16, 17]. A common theme in these and other studies has been to quantify the effect of friction [17, 7, 12] or optimal lubricant [4, 6, 12, 13] and barreling [5, 11, 16, 9]. on interpreting the stress-strain behavior of the test specimen.

ASTM International standard test method E9, [2] first established in 1924, is a consensus standard for conducting compression tests to establish the strength of materials. It contains methods for testing cylindrical test specimens as well as methods for testing sheets with lateral support. The standard contains requirements for calibrating and qualifying the testing machine including extensometers, and aligning the fixtures and test specimen. It recommends, but does not require, specific test specimen geometries and lubricants. In addition, it suggests several fixture designs. It does not require the use of an extensometer in contact with the test specimen to measure strain, and most studies that reference E9 infer the test specimen strain and stress from the displacement of the actuator.

Although the first version of E9 was released eight decades ago, its precision has never been formally evaluated, as required [19] Such evaluations require a formal interlaboratory study, which can benefit both end user and testing laboratories. Users need the results of interlaboratory tests to determine the uncertainty that should be associated with the value of a material parameter obtained using a test method. Laboratories that employ test methods use interlaboratory studies to identify the deficiencies in and improve their implementation of a test method.

This manuscript reports the results of an interlaboratory study to establish the precision of ASTM International standard test method E9 for determining the yield strength and elastic modulus in compression. The results of this study were incorporated into the Precision and Bias statement of E9 in 2009. This report goes beyond the research report [20] that documents the calculation of the precision by comparing the results to other interlaboratory studies of uniaxial test methods, Sec. 4.1, analyzing some of the possible sources for the variability,Sec. 4.2, and presenting a method to evaluate the elastic modulus measured in the test, Sec.4.3.

Table 1: Summary of literature that analyzes the compression test.

Reference	Content	$e_{\max}$	Materials	Lubricant	Notes
Banerjee, 1985[5]	b	1.6	Al	teflon, MoS_2, oil, none	
Carter, 1985[6]	F	0.35	Al	MoS_2, none	
Chait, 1975[4]	r				review article
Cook, 1945[7]	f	0.7	Cu	none	change friction via platen surface finish
Gunasekera, 1982[8]	f	1.2	1022 steel	teflon	
Hsü, 1969[9]	bfF		Cu	teflon	
Kamaluddin, 2007[10]	F	0.8	Al	grease	
Kobayashi, 1970[11]	b	1.4	1040 steel	graphite	
Lovato, 1992[12]	f	1	Al, Nb, brass, steel	MoS_2, teflon, BN, none	grooved platens
Male, 1966[13]	f	0.8	Al, Ti, brass	graphite, lanolin, paraffin	
Mescall, 1983[14]	F		4340 steel		
Ray, 1983[15]	f		steel	teflon	method for correcting for aspect ratio
Schey, 1982[16]	F	1	1020 steel, 6061 Al	MoS_2, teflon	
Woodward, 1977[17]	f	1.2	steel	teflon	method for correcting for aspect ratio

$e_{\max}$: maximum strain in test
Key to content of reference
b: barreling analysis
f: friction analysis
F: finite element analysis
r : review article

2 Experimental Procedure

This interlaboratory study followed the methods of ASTM E691 [21], and uses statistical terms in accord with ASTM E177 [22].

2.1 Participants

Using the ASTM International [23] and American Association for Laboratory Accreditation [24] laboratory directories, the organizers contacted and discussed the interlaboratory study with twenty-five possible laboratory participants. From this original list thirteen laboratories agreed and were able to participate, and ten ultimately completed

Table 2: List of participants in this study.

Participant	URL
AADFW, Inc, Euless, Tx	http://www.aadfwinc.com
Alcoa, Pittsburgh, Pa	http://www.alcoa.com
Exova, Glendale Hts, IL	http://www.exova.com
Dickson Testing Company Inc., South Gate, Ca	http://www.dicksontesting.com/
Imperial College Mechanical Engr., London, England	http://www.imperial.ac.uk/
MAR-TEST, Inc. (Cincinnati), Cincinnati, Oh	http://www.mar-test.com/
MAR-TEST, Inc. (Stuart), Stuart, Fl	http://www.mar-test.com/
Metcut Research Inc., Cincinnati, Oh	http://www.metcut.com
Stork Climax Research Services, Wixom, MI	http://www.storksmt.com/crs
Westmoreland Mechanical Testing, Youngstown, Pa	http://www.wmtr.com

the test program, Table 2.

2.2 Instructions and method

The participants followed ASTM Standard Test Method E9 [2] to establish the elastic modulus, E, and 0.2 % offset yield strength, *YS*(0.2 % offset). At the time of the study, the version of E9 in was E9-89a, but the only non-editorial difference between E9-89a and the current version, E9-09, was the addition of the precision statement to the latter, which was the purpose of the interlaboratory study. The participants also returned electronic traces of the stress-strain curves to the organizers. All compression fixtures were required to be qualified according to ASTM E9 [2] Section 6.6 using at least five of test specimens supplied, unless the participant had already qualified the test setup according to Section 6.6. The participants conducted the compression tests using at least one extensometer at a nominal strain rate of $de/dt = 0.005$ min^{-1} $= 8.33 \times 10^{-5}$ s^{-1}. Each participant reported ten items as required in sections 10.1.1–10.1.9 and 10.1.13 of standard method E9: Material (test specimen ID), configuration description, test specimen dimensions as tested, fixture and lubricant description, testing machine description, speed of testing (required in section 8.7; report actual value), stress-strain diagram, modulus of elasticity, E, Yield strength, *YS*(0.2 % offset), and any anomalies.

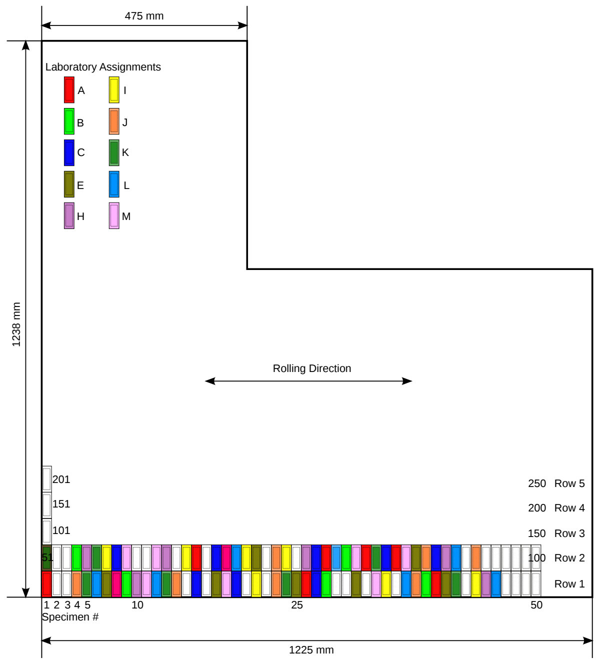


Figure 1: Schematic of the location of test specimen blanks in the original plate and a drawing of the finished compression test specimen.

2.3 Test Specimen

The material tested was aluminum alloy AA2024-T351, which is solution heat-treated and stress-relieved by controlled stretching. It was supplied as a plate with thickness, $t = 22.2$ mm (0.875 in), from which the organizers cut and distributed test specimen blanks, which were distributed throughout the plate. Figure 1 describes the location of the test specimens in the original plate and their numbering scheme. Only test specimens in rows 1 and 2 were used in this study. Test specimens number 1-50 came from row 1; test specimens 51–100 came from row 2. Figure 2 shows the test specimen with dimensions and tolerances. The drawing provided to the participants showed the dimensions in non-SI units. Participants machined their own test specimens from sawed blanks that the organizers supplied.

Test specimens were tested with the loading axis transverse to the rolling direc-

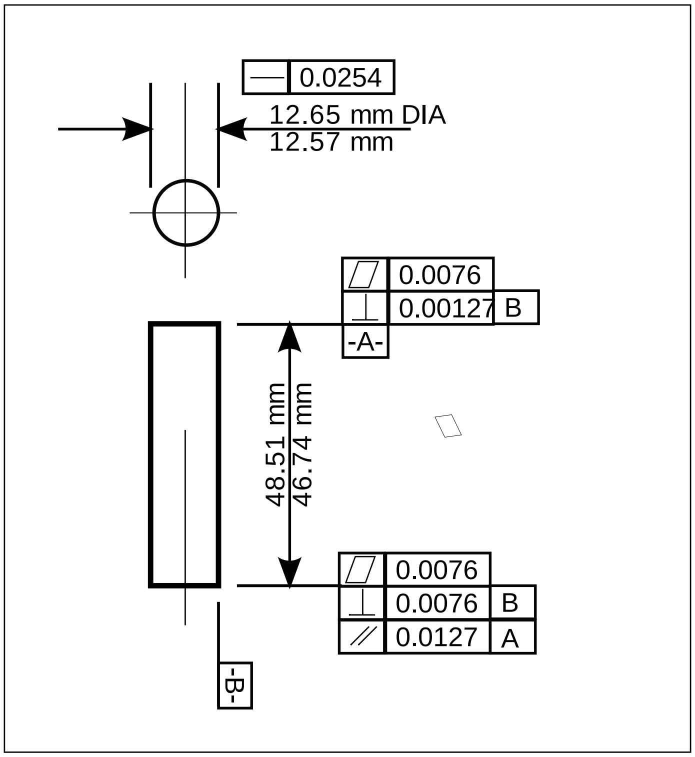
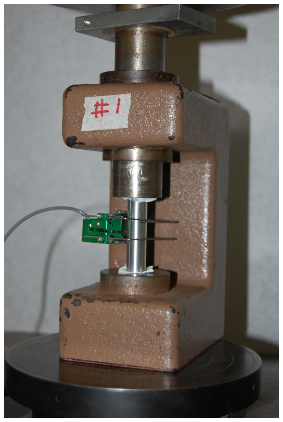


Figure 2: Dimensions of the test specimen used.

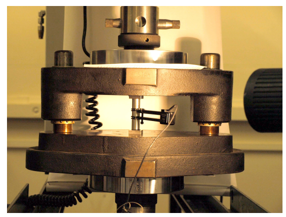
tion in the plane of the plate, the so-called long-transverse ("LT") orientation, Mil-Handbook-5J [25] Figure 1.4.12.3(a). The test specimen ID takes the form "LT-NN-L-X" where "LT" identifies the orientation relative to the rolling direction, "NN" identifies the test specimen number (1–250), "L" identifies the laboratory that received the test specimen blank (A-K) and "X" identifies the test order of the test specimen at a given laboratory.

2.4 Compression fixtures

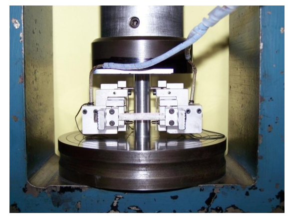
Figure 3 shows, in no particular order, some of the compression fixtures that the participants used. Table 3 describes the alignment capability, number of extensometers, and the lubricant used in each laboratory. The participants used a variety of loading fixtures, some of which involved a sub-press mounted in the testing machine to improve alignment. Others used adjustable platens that were aligned and then locked. Three laboratories used diametrically opposed extensometers instead of a single extensometer. No laboratory reported strain from actuator displacement.



(a)



(b)



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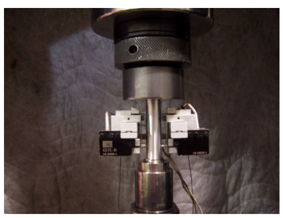

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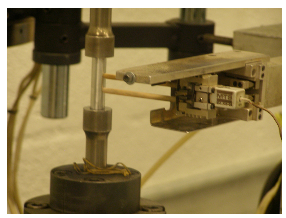
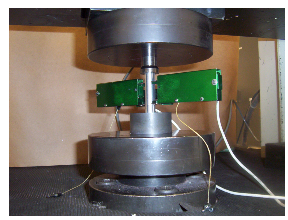


(e)



(f)

(g)

Figure 3: Some compression fixtures used in this study.

3 Results

3.1 Stress-strain behavior

Figure 4 plots the engineering stress-strain curves by laboratory. In each case the stress-strain curve was shifted along the strain axis so that a linear fit to the stress-strain $(S-e)$ data in the range $(50 < S < 175)$ MPa intercepts the origin. Figure 5 shows four views of the same stress-strain curves on a single plot.

Table 4 summarizes the reported modulus, E, and yield strength, *YS*(0.2 % offset), for the laboratories. Figure 6 plots the reported elastic modulus, E, by laboratory. Figure 7 plots the reported 0.2 % offset yield strength, *YS*(0.2 % offset), by laboratory.

Table 3: Descriptions of the compression setup, extensometers and gauge lengths, G, and lubricants for all laboratories.

Lab	test setup	Extensometer	Lubricant
A	No sub-press	Two, opposed, class B-1 $G = 25.4$ mm	Not reported
B	Sub-press	Single, class not reported $G = 12.7$ mm	Molybdenum disulfide
C	No sub-press, no spherical seat	Single, class not-reported $G = 25.4$ mm	None
E	sub-press	Single, class B-1 $G = 25.4$ mm	Teflon tape
H	No sub-press	Single class B-2, $G = 12.7$ mm	Molybdenum disulfide
I	No sub-press	Two opposed, class B-2, $G = 25.4$ mm	Not reported
J	Precision ground sub-press, aligned to closer tolerance than required for specimen, no spherical seat	Two opposed, class B-1 $G = 25.4$ mm	None
K	No sub-press. Platens aligned, shimmed and then locked. No spherical seat	Single, class B-2, $G = 12.7$ mm	Molybdenum disulfide
L	Sub-press	Single, class B-2 $G = 25.4$ mm	WD-40
M	Compression platens mounted in aligned hydraulic grips	Single, class B-2, G not reported	None

3.2 Testing rates

Figure 8 plots the strain as a function of time for the laboratories that reported time data. The interlaboratory instructions did not specify a control mode for the test. Four laboratories (C,H,K,L) conducted the test in strain control from the extensometer signal, as indicated from the constant slope of the strain-time plot, Figure 8. Three labora-

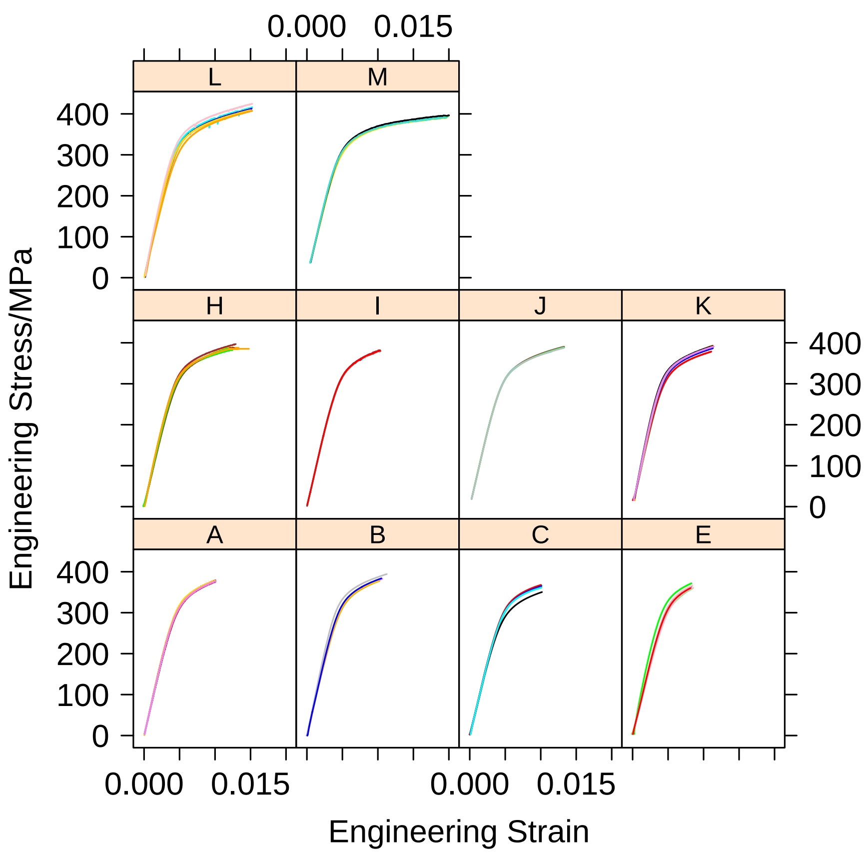


Figure 4: Engineering stress-strain curves.

tories (A,E,J) conducted the test in position control, as indicated from the changing slope in the strain-time plot. Figure 9 plots the strain rates in the elastic and plastic portions of the stress-strain curves calculated by linear regression. The plastic strain rate was calculated for strains greater than the strain at the 0.2% offset yield strength: $e_{\text{YS002}} < e < e_{\max}$. The elastic strain rate was calculated in the range $0 < e < (e_{\text{YS002}} - 0.002)$. Laboratory J conducted the test in position control and set the elastic strain rate to the specified value.

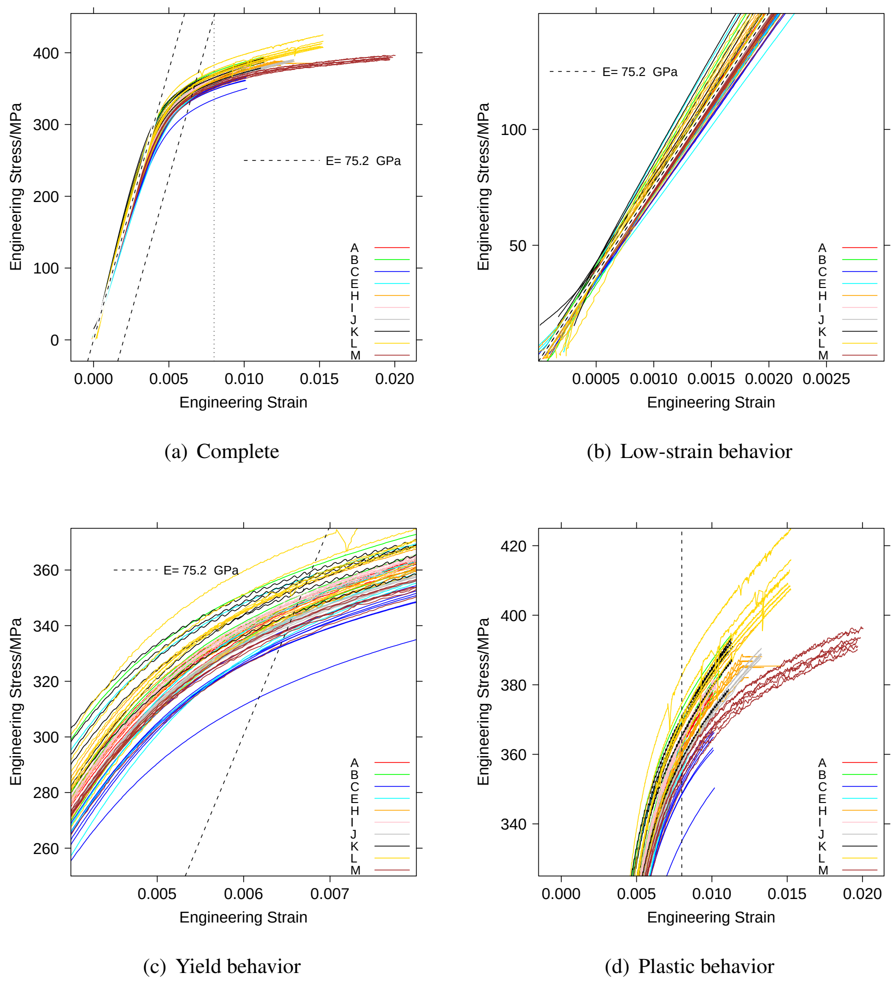


Figure 5: Engineering stress-strain curves: (a) complete behavior, (b) low-strain behavior, (c) yield behavior, and (d) plastic behavior. Dashed lines show the accepted value for the modulus of 2024-T351, $E = 75.2$ GPa, the 0.2 % offset yield strength determination, and the $e = 0.008$ total elongation.

4 Analysis and discussion

4.1 Expected variability

Table 5 summarizes the mean and standard deviation, sd, and coefficient of variation, cv, of the reported n measurements of the elastic moduli and yield strengths. Table 6 defines the statistical parameters used in this section.

Table 4: Reported data

Lab	Sample	*E*	*YS*	Lab	Sample	*E*	*YS*	Lab	Sample	*E*	*YS*
A		75.6	339	H		77.2	345	K		75.8	343
A		72.9	340	H		73.8	347	K		80.7	348
A		76.8	341	H		73.1	347	K		87.6	351
A		75.1	342	H		77.2	347	K		84.1	351
A		73.8	342	H		75.8	347	K		80	352
A		74.7	342	H		77.2	347	K		76.5	354
A		76.3	343	H		78.6	354	K		NA	NA
B		73.1	348	I		75	348	L		73	348
B		76.5	349	I		76	349	L		84.2	349
B		75	349	I		75	349	L		78.4	352
B		75	353	I		76	350	L		84.2	355
B		83.4	356	I		75	350	L		75.7	356
C		72	318	I		75	350	L		77.5	356
C		72.8	335	I		76	351	L		85.8	363
C		71.5	336	J		74.5	340.5	M		69	338
C		72.4	338	J		74.2	341.6	M		72	342
C		71.4	338	J		74.4	341.6	M		71	342
C		70.7	339	J		74.5	342.1	M		70	342
C		72.9	341	J		74.8	342.5	M		72	342
E		68.3	342.2	J		74.8	343.1	M		72	343
E		67	346.8	J		74.1	343.4	M		68	345
E		69.3	347.4								
E		68.6	349								
E		70.5	351.1								
E		70.9	351.4								
E		70.6	355.8								

YS=*YS*(0.2 % offset)

The results of an interlaboratory study contain variability that arises within a given laboratory and variability that arises between laboratories. The terms repeatability and the reproducibility, denoted by subscripted "r" and "R," are often used to differentiate between the sources [22]. In a general sense, repeatability characterizes the ability of an individual laboratory to repeat measurements, while reproducibility characterizes the ability of an individual laboratory to achieve the global mean or accepted value. For example, if the results from an individual laboratory are tightly grouped, their repeatability is high, but their mean value may still deviate significantly from the accepted value, in which case their reproducibility is low.

Both the variability in the material and the variability of the test method influence the repeatability. The excellent repeatability of the measurements for both E and *YS*(0.2 % offset) in laboratories A, I, and J , Table 5, shows that the material variability in this study is quite low, and therefore the implementation of the test method by the individual laboratories is the major contributor to repeatability.

Figure 10 and Table 7 compare the results for repeatability and reproducibility for

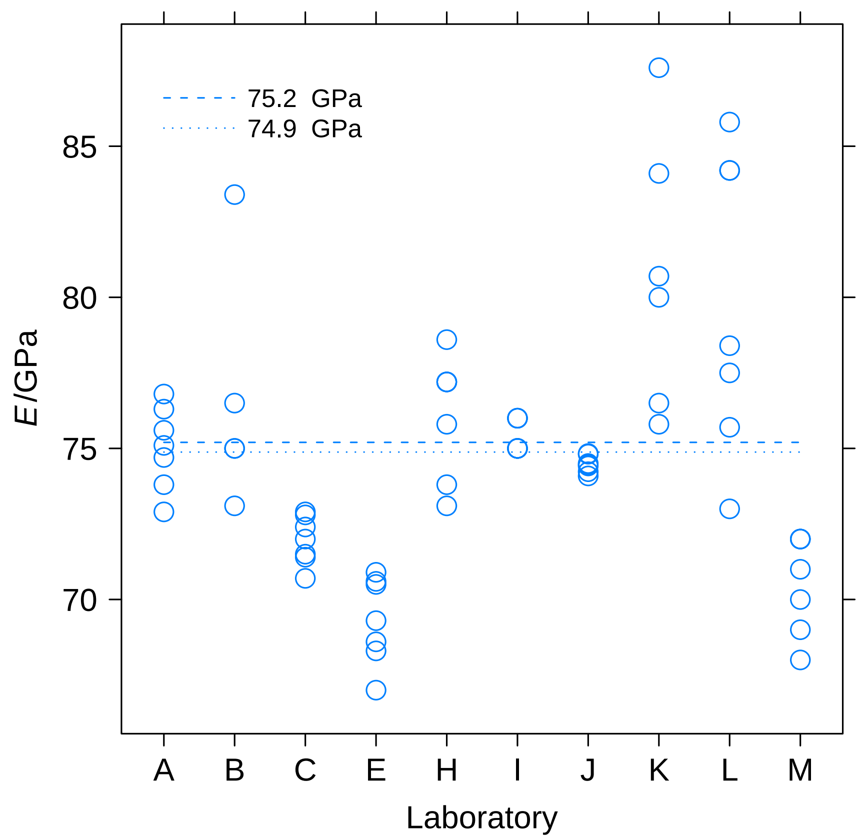


Figure 6: Reported elastic modulus, E. Dashed lines show the average reported modulus and a commonly accepted value of the modulus (E=75.2 GPa) from Mil-HDBK-5J [25].

this study to the results of other interlaboratory studies of tensile tests at room [26, 27] and elevated temperatures [28] to establish the 0.2 % offset yield strength, *YS*(0.2 % offset). In Figure 10 the within-laboratory and between-laboratory standard deviations, s_r (Eq. 4) and s_R Eq. 7, see Table 6, are divided by the mean value and expressed as their respective coefficients of variation, cv_r, Eq. 5 and cv_R, Eq. 8.

The repeatability coefficient of variation of the yield strength measurements in this study, derived from the average of the standard deviations of the individual laboratories, is cv_r = 0.0111 Eq. 5, while the coefficient of variation of the reproducibility, cv_R = 0.0197 Eq. 8, is about twice as large. The repeatability and reproducibility of compression tests established in this study are among the best measurements of all reported uniaxial measurements [26, 27, 28].

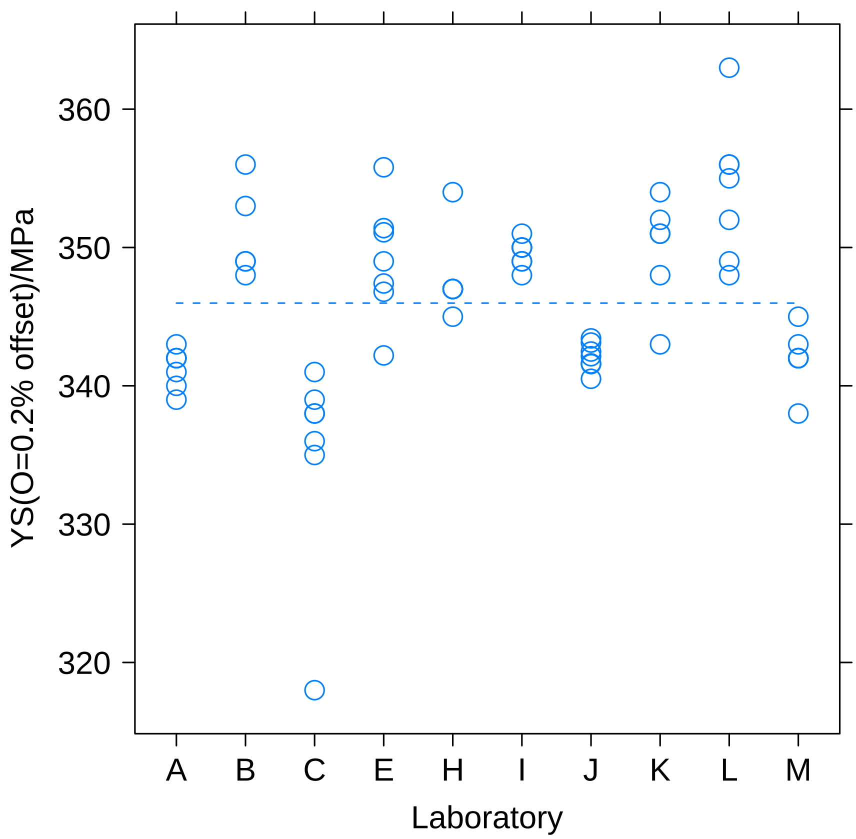


Figure 7: Reported 0.2 % offset yield strength, *YS*(0.2 % offset). The dashed line is the average of the results from all laboratories.

4.2 Effect of test method implementation

Because the laboratories were free to implement the test method in different ways, while still complying with the standard, their result can be used to learn about the effect of different aspects of test method on the measurement. In particular, the results can be examined to determine the effects of the number of extensometers and lubrication on the quality of the measurement.

4.2.1 Effect of number of extensometers

Some laboratories used a single extensometer, while others used two opposed extensometers to measure the strain. Figure 11 plots the reported modulus and yield strength for the laboratories, grouped by the number of extensometers used in the determination.

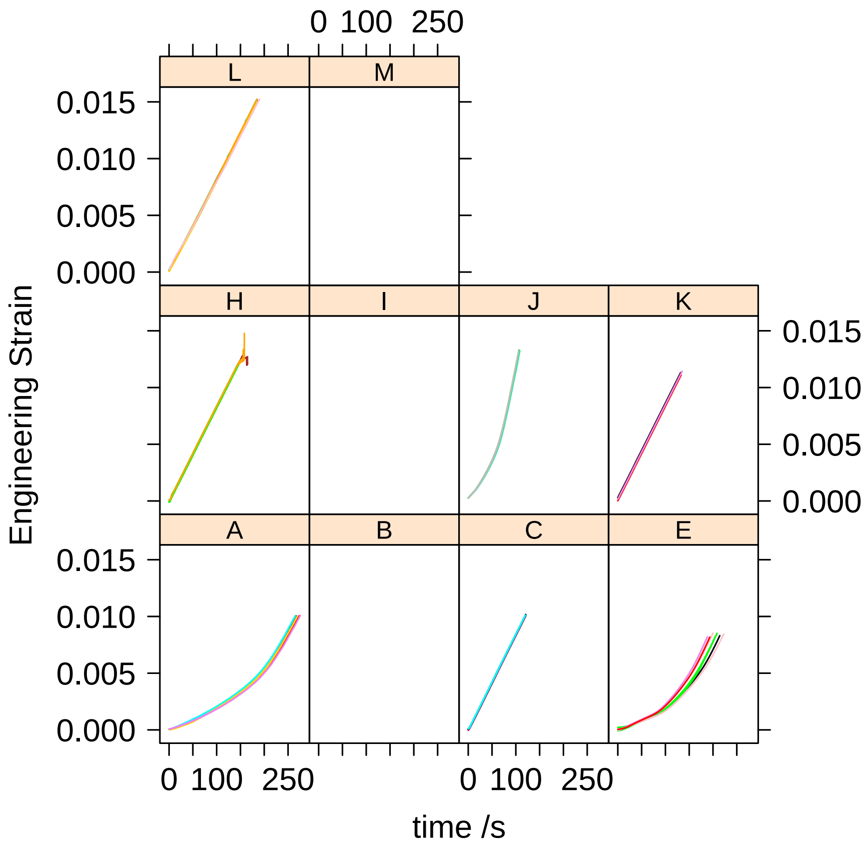
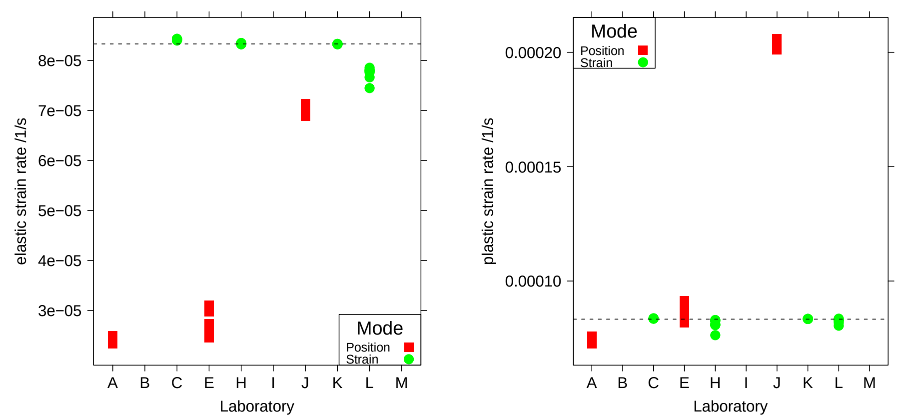


Figure 8: Reported strain as a function of time.

The three laboratories, A, I, J, with the best repeatability and reproducibility of E and *YS*(0.2 % offset) are the only ones that used a system of two-opposed extensometers instead of a single extensometer. In addition, the measured elastic modulus in these three laboratories was the closest to the accepted [25] value $E = 75.2$ GPa, Figure 11a. Note that the repeatability of the modulus and yield strength from the labs that used a single extensometer is worse (i.e. larger variability), but also that for an individual laboratory, the values usually all lie above or below the global average value. Using two extensometers will tend to average the effect of non-axial loading, since during bending, one side of the test specimen will be displaced more, and the other less. That the values for an individual, single-extensometer laboratory are usually displaced to one side of the global mean value points toward the alignment of the compression fixture, rather than machining of the test specimen as the source of the non-axiality. If the ends of the test specimen were not parallel, the measured values would tend to encompass the mean



(a) elastic strain rate (b) plastic strain rate

Figure 9: Calculated (a) elastic and (b) plastic strain rates for laboratories that reported time data. Symbols differentiate between position-control and strain-control tests. Dashed line indicates the requested rate, $de/dt = 8.33 \times 10^{-5}$ 1/s.

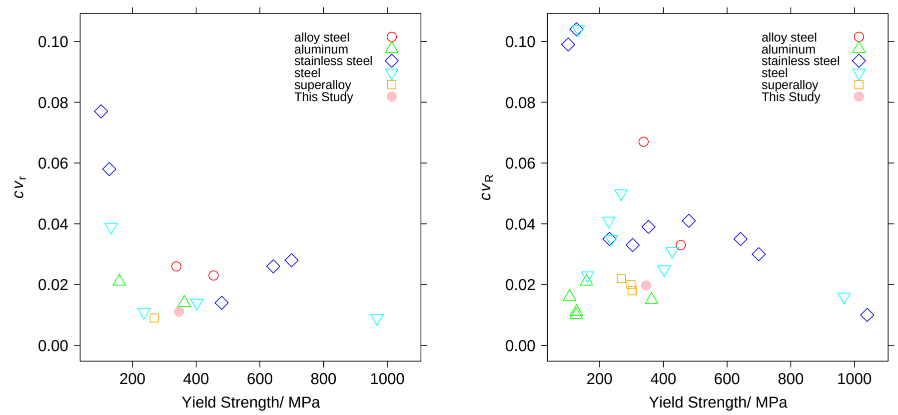
Table 5: Summary statistics for the modulus and yield strength data.

Lab	n	$\bar{E}$ GPa	sd(E) GPa	$cv(E)$	$\bar{YS}$ MPa	sd(YS) MPa	$cv(YS)$
A	7	75.0	1.4	0.018	341.3	1.4	0.004
B	5	76.6	4.0	0.052	351.0	3.4	0.010
C	7	72.0	0.8	0.011	335.0	7.7	0.023
E	7	69.3	1.4	0.021	349.1	4.3	0.012
H	7	76.1	2.0	0.026	347.7	2.9	0.008
I	7	75.4	0.5	0.007	349.6	1.0	0.003
J	7	74.5	0.3	0.004	342.1	1.0	0.003
K	7	80.8	4.5	0.056	349.8	3.9	0.011
L	7	79.8	4.9	0.062	354.1	5.1	0.014
M	7	70.6	1.6	0.023	342.0	2.1	0.006

Note: *YS*= *YS*(0.2 % offset)

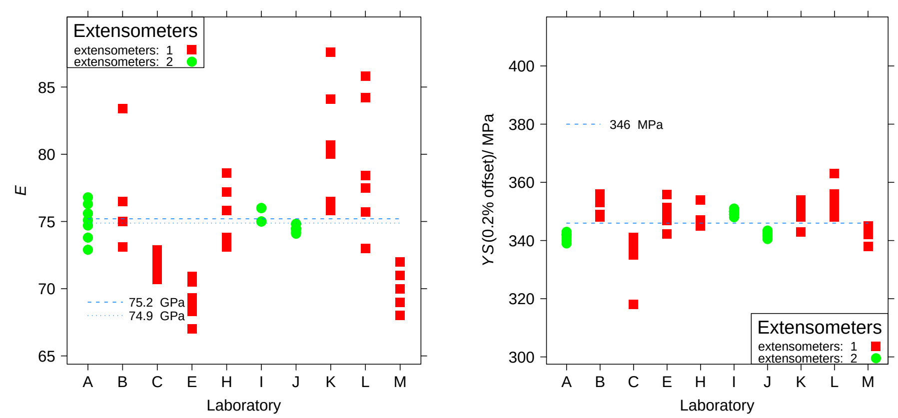
Parameter	Grand Average	s_r	cv_r	s_R	cv_R
E	$\bar{\bar{E}}$= 75.0 GPa	2.7 GPa	0.036	4.4 GPa	0.059
YS(0.2 % offset)	$\bar{\bar{YS}}$(0.2 % offset)= 346.2 MPa	3.8 MPa	0.011	6.8 MPa	0.020

Table 6 defines the parameters. Because laboratory B tested only five specimens instead of seven, the mean values shown in Figures 6, 7, 11, and 13 differ from the Grand Average, also known as the average of cell averages, Table 6, Eq.(2).



(a) Repeatability (b) Reproducibility

Figure 10: Literature data [26, 28, 27] on (a) repeatability (within-laboratory) and (b) reproducibility (between-laboratory) of yield strength, *YS*(0.2 % offset). Table 7 summarizes the data.



(a) Modulus (b) *YS*(0.2 % offset)

Figure 11: Reported (a) modulus and (b) 0.2 % offset yield strength, *YS*(0.2 % offset), identified by number of extensometers used.

value, because no special relationship exists between the test specimen and the loading fixture. Conversely, the extensometer is usually mounted on the test specimen with a fixed relation to the orientation of the subpress or loading platens, so a slightly misaligned loading fixture is always in the same geometric relation to the extensometer, which will tend to alway over- or under-estimate the strain.

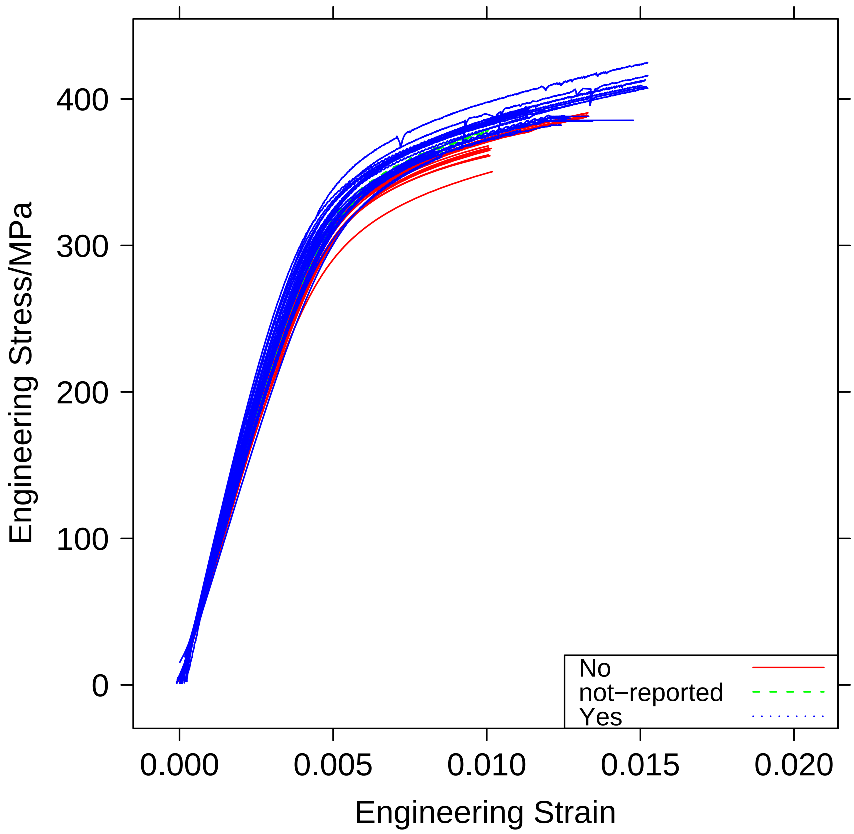
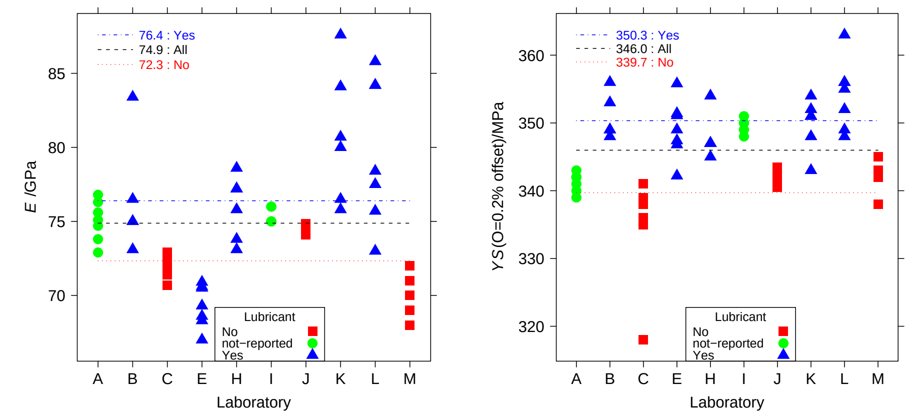


Figure 12: Effect of lubricant on stress-strain behavior.

4.2.2 Effect of Lubricant

The standard allows the laboratory to choose whether to lubricate the ends of the test specimen during the test. Figure 12 shows the engineering stress-strain curves, identified by the use or omission of lubricants. Several laboratories did not report lubricant use. Table 3 identifies the lubricants used. Three laboratories used no lubricant, and their stress-strain curves are the lowest in the collection. Figure 13 plots the reported modulus, E, and yield strength, *YS*(0.2 % offset), identified by lubricant use. The average yield strength from the three laboratories that did not use lubricant was 10.6 MPa lower (3.1 %) than the average of the laboratories that lubricated the specimen. Similarly the average reported elastic modulus, E, from the three laboratories was 4.1 GPa (5.7 %) smaller. Table 8, an analysis of variance, shows that the reported yield strength, *YS*(0.2 % offset), and modulus, E,are both significantly different for the lubricated and unlubricated cases.



(a) Modulus, E

(b) Yield strength, *YS*(0.2 % offset)

Figure 13: Effect of lubricant on (a) modulus E and (b) yield strength *YS*(0.2 % offset) . Dashed lines are the mean values for all data, including "not-reported," not lubricated ("No") and lubricated ("Yes") tests.

Since most laboratories used an extensometer with a gauge length $G = 25.4$ mm on the test specimen, 2, with length $l \approx 48$ mm and diameter $d = 12.7$ mm, the end of the test specimen is approximately one test specimen diameter away from the extensometer contact point. That distance complies with the requirements of E9, and should minimize the effect of the non-uniform stress caused by friction at the specimen ends. No study has examined the sensitivity of the strain measurement to the non-uniform stress at the end of the specimen, but the existence of non-uniform stresses caused by friction is well known [29]. The origin of the difference remains unexplained.

4.3 Elastic Modulus

The elastic modulus measured in the test is very sensitive to the alignment and sensitivity of the test setup. As a result, ASTM E 9 [2] requires that laboratories validate their compression test fixture by measuring the elastic modulus of aluminum alloy 2024-T4 bar, according ASTM E 111 [30] and obtaining measurements on five consecutive specimens to within 5 % of a stated value, E=73.8 GPa. The behavior of the reported elastic modulus should be a good indicator of the quality of the compression test facility.

The interlaboratory study did not require laboratories to revalidate their test setups, but they did report the measurements of elastic modulus, Figure 6, made during the compression tests. Most of the laboratories only used a ASTM E 83 [31] Class B-2 extensometer, see Table 3, rather than the roughly twice-as-accurate class B-1 extensometer required by ASTM E 111 [30] when elastic modulus is the primary parameter to be determined. But because the primary parameter to be determined was the yield strength, E 9 Section 9.2 permits the use of the class B-2 extensometer.

Many of the following plots evaluate the quality of the measurements by employing the absolute modulus error, ΔE, in a measurement, which is defined as the absolute value of the difference between the accepted modulus [25] for 2024-T351 aluminum, $E_{\text{Al}}^{\text{acc}} = 75.2$ GPa and the calculated modulus, E.

$$\Delta E = |E_{\text{Al}}^{\text{acc}} - E| \tag{9}$$

Other references [32, 33, 34, 2] recommend different values in the range 73.8 GPa $<$ $E <$ 76.1 GPa, and differentiate [33] between longitudinal and transverse orientation.

To reduce the variability due to the interpretation of the individual laboratory, and to put each modulus measurement on a consistent basis, we reanalyzed each stress-strain curve to estimate the elastic modulus using a technique based on the method of Scibetta and Schuurmans [35]. In addition to estimating the modulus, the method includes some checks on the quality of the data that may be useful for laboratories that seek to improve their modulus measurement.

Method for analysis The method consists of nine steps.

1 Find the knee of the stress-strain curve by the method of Scibetta and Schuurmans [35].

   1. Find the point on the stress-strain curve (x_1, y_1) closest to $y_1 = 0.05\max(y)$
   2. Create point (x_o, y_o) where $x_o = x_1$ and $y_o = 0.2\max(y)$.
   3. Find point (x_t, y_t) which is the point on the test record an on the tangent drawn from (x_o, y_o).

2 Truncate the stress-strain data at (x_t, y_t) and normalize by these values. Retain the maximum values of the test record: $(x_{\text{max}}, y_{\text{max}})$.

3 Check digital resolution, δ, of stress and strain. The digital resolution should be $\delta < \frac{3}{2^{12}} \frac{x_{\text{max}}}{x_t}$. No more than more than 25 % of points should have zero stress or strain change from the previous point.

4 Use the Scibetta-Schuurmans [35] algorithm to determine optimum fit window and calculate the slope, which is the elastic modulus E. The Scibetta-Schuurmans algorithm finds the optimal region for determining the modulus by performing a linear regression on every possible subset of data in the region $x_o < e < x_t$ that contains at least 20 % of the data. The regression with the lowest residual standard deviation is the optimum fit. In some cases, more than one million fits were evaluated for each stress-strain curve.

5 Check for excessive noise in optimum fit window, defined as the standard error of the fit: s_j defined in Table 6 where n is the number of stress-strain pairs in the optimal region, Y_{ij} is the stress evaluated at point j, and $\bar{Y_j}$ is the predicted stress from the fit to the optimal region. Repeat the regression of strain upon stress as well. In both cases, the standard error in strain or stress should not be greater than 0.01.

6 Extend the range of fit to include all stress points whose deviation from the optimal fit line is less than one times standard error computed in Step 5.

7 Refit the extended data set to determine modulus, E.

8 Examine the shape of the stress and strain residuals as a function of strain in the extended fit. If the slope of the residuals in the first or fourth quartile of the extended range is more than 0.05, the data exhibit excessive curvature. At least five points must exist in each quartile to evaluate this curvature.

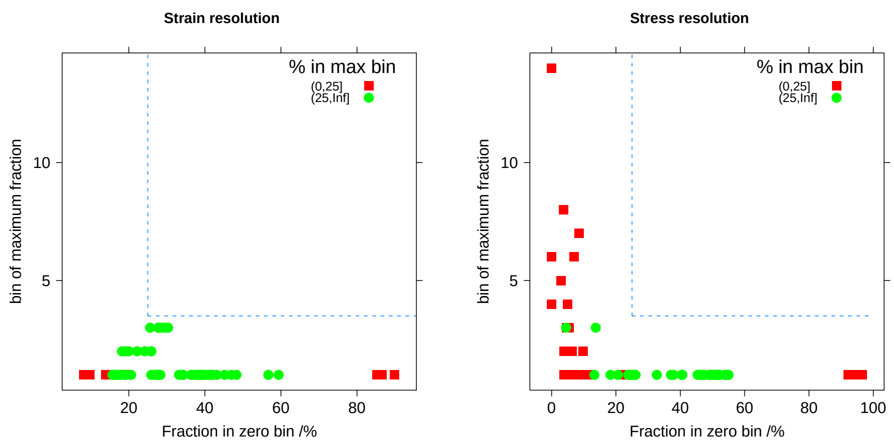
9 Check that the optimal fit range is greater than $0.4y_t$.

The method employs four quality metrics:

- Data quality 1: digital stress and strain resolution should be sufficient (Step 2)
- Data quality 2: strain or stress signal should not have excessive noise (Step 5)
- Fit quality 1: the stress and strain residuals in extended data range should not have excessive curvature (Step 8)
- Fit quality 2: the optimal fit range should be greater than or equal to 40 % of the total data range (Step 9)

Data Quality 1: sufficient digital resolution Figure 14 summarizes the strain and stress resolution. To create the figure, the digital resolution was estimated from the data set and binned into a histogram. The figure plots the index of the bin with the maximum fraction of the data, in units of δ, against the fraction of the points in the "zero" bin, which is the fraction of points where the stress or strain value did not change between readings. The symbols show the fraction of the points in the bin of maximum fraction, broken into two groups: those where the bin of maximum fraction contained less than 25 % of the data ($(0, 25]$) and those that contained more than 25 % of the data ($(25,$ Inf). The method identifies experiments with insufficient digital resolution as those where the bin number of maximum fraction is greater than 3 *and* either the fraction in the zero bin is > 25 % *or* the fraction in the bin of maximum fraction is > 25 %. The first condition is identified by the dashed lines. The second "or" condition comprise those points identified by green circles with y value greater than 3. No experiment demonstrated insufficient digital resolution.

Data Quality 2: excessive noise Excessive noise in the strain or stress signal will degrade the quality of the measured modulus. The method identifies experiments with excessive noise in these signals as ones in which the standard error of the fit is greater than 0.01. Figure 15 plots the standard error of the fit for both strain and stress by laboratory. The symbols identify three levels of modulus error, Eq. 9. The noise in all the experiments was sufficiently low. No obvious relation exists between the absolute modulus error, ΔE, and either the standard error of stress or strain.



(a) strain resolution (b) stress resolution

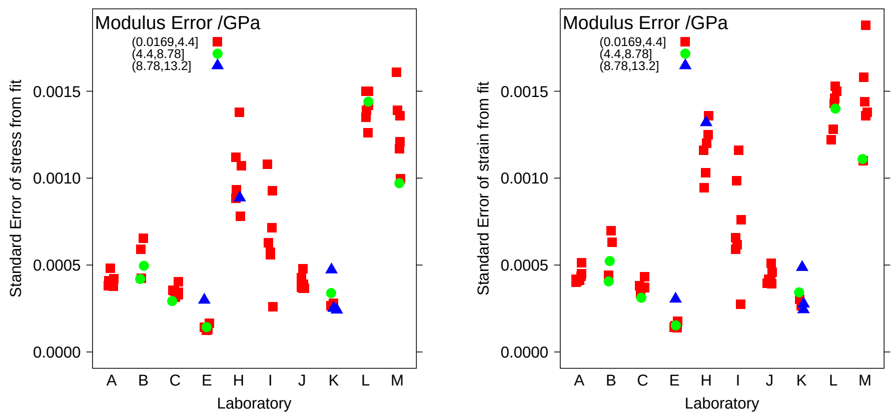
Figure 14: (a) Strain and (b) stress data resolution. Dashed lines enclose the region of insufficient resolution. Symbols denote the fraction of points in the bin of maximum fraction.

Fit Quality 1: Excessive curvature in extended data set Some metric of the curvature of the optimal fit range is necessary, since the method only selects the best fit over an optimal region. That fit might still by poor. One method for examining the quality of the fit is to examine the deviation from the fit line in the first and fourth quartiles of the optimal range. Curvature of the stress-strain record frequently appears this way. If the slope of the residuals vs. strain in the outer quartiles is greater than 5 % of the slope in the optimal region, the curvature of the fit is excessive.

Figure 16 shows the slope of the residuals in the outer quartiles. Different symbols show the level of absolute error of the elastic modulus, Eq. 9. All but one of the tests fall inside the ± 5 % limits. In addition, linear regression of the absolute modulus error against the absolute value of the residual slope reveals no relationship.

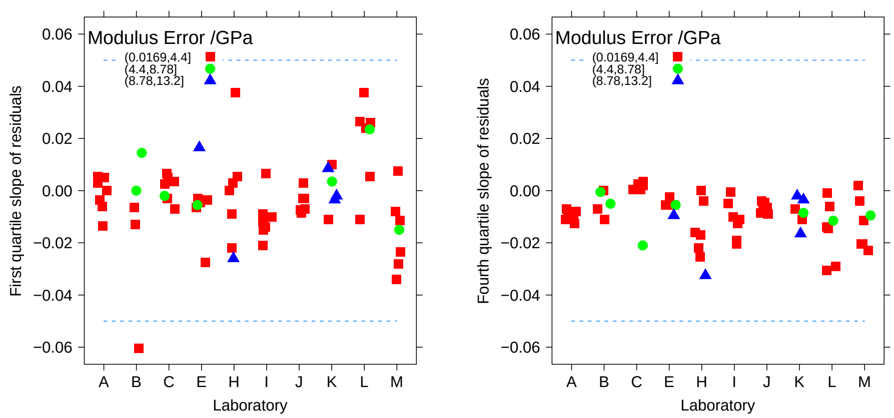
Fit Quality 2: Final fit range The second measure of the quality of the fit to determine the elastic is the size of the range, R_f, of the optimal fit. Figure 17 plots the absolute modulus error, Eq. 9, against the fraction of the range of original stress data. The dashed line is the boundary of the minimum acceptable limit $R_f \geq 0.4$. The absolute modulus error, Eq. 9, increases as the size of the fit region decreases, as expected, and the boundary between the two regions lies at the chosen final fit range minimum $R_f = 0.4$. The mean error for tests with fit region $R_f < 0.4$ is 2.5 times as large (4.88 GPa vs. 1.95 GPa) as that from the acceptable region.

Conclusions from elastic modulus analysis Of the quality metrics, the size of the final fit range is the best predictor of overall quality of the modulus measurement, and the level chosen, $R_f \geq 0.4$, is a good metric for identifying potential problems with



(a) strain noise (b) stress noise

Figure 15: RMS noise in calculated modulus based on (a) strain, and (b) stress. Symbols denote three increasing levels of absolute modulus error, Eq. 9. All measurements are below the acceptance limit of 0.01. Data have been jittered to prevent overplotting of points.



(a) 1st quartile slope (b) 4th quartile slope

Figure 16: Slope of residusals in the (a) 1st, and (b) 4th quartile of the extended fit for modulus . Symbols denote three increasing levels of absolute modulus error, Eq. 9. Dashed lines denote the acceptance limit Data have been jittered to prevent overplotting of points.

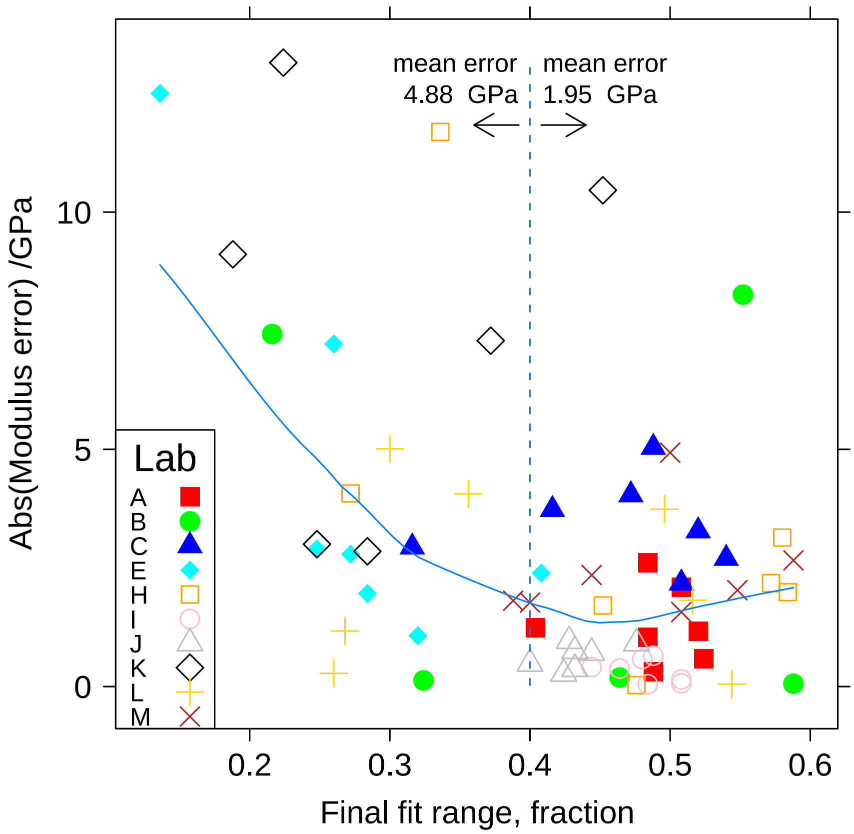


Figure 17: Absolute error in modulus, Eq. 9, as a function of the fraction of the range, R_f, used to calculate the modulus. Symbols identify the different laboratories. Points to the left of the dashed line are deemed to have insufficient fit range.

modulus measurement.

5 Conclusions

Five conclusions can be drawn from this study.

- The repeatability of yield strength determined from compression tests using ASTM E9 can be expected to be about 1.1 % of the mean value, cv_r= 0.011 , Figure 7 and Table 5.

- The reproducibility of yield strength determined from compression tests using

ASTM E9 can be expected to be about 2 % of the mean value, cv_R= 0.020 , Figure 7 and Table 5.

- Despite the perceived difficulties with alignment in compression, the repeatability and reproducibility of the compression test was among the best measured for uniaxial tests, Figure 10
- Using two diametrally opposed extensometers instead of a single extensometer can improve the precision of the strain measurement by more than two times, Figure 11
- If the final fit range for estimating the modulus is less than 40 % of the elastic region, the modulus measurement is frequently in error, Figure 17

Author contributions

William Luecke supervised interlaboratory study and analyzed the reported modulus and yield strength data. Stephen Graham and Michael Adler analyzed the stress-strain curves to recalculate the modulus and the associated data and fit quality.

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Certain commercial equipment, instruments, or materials are identified in this paper in order to specify the experimental procedure adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose.

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Table 6: Definitions of repeatability and reproducibility statistics.

Formula	Description
n	number of tests in a laboratory, $n \approx 7$
p	number of laboratories, $p = 10$
Y_{ij}	individual test result in lab j
	$1 < i < n \quad ; \quad 1 < j < p$
$\bar{Y}_j = \frac{1}{n}\sum_{i=1}^{n} Y_{ij}$	average test result in lab j (cell average) (1)
$\bar{\bar{Y}} = \frac{1}{p}\sum_{j=1}^{p} \bar{Y}_j$	average of cell averages (grand average) (2)
$s_j = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n}(Y_{ij} - \bar{Y}_j)^2}$	standard deviation measured in lab j (3)
$s_r = \sqrt{\frac{1}{p}\sum_{j=1}^{p}(s_j)^2}$	repeatability (within-lab) standard deviation (4)
$cv_r = \frac{s_r}{\bar{\bar{Y}}}$	coefficient of variation within laboratories (5)
$s_{\bar{Y}} = \sqrt{\frac{1}{p-1}\sum_{j=1}^{p}(\bar{Y}_j - \bar{\bar{Y}})^2}$	standard deviation of cell averages (6)
$s_R = \sqrt{s_{\bar{Y}}^2 + s_r^2(\frac{n-1}{n})}$	reproducibility standard deviation ((between-lab) (7)
$cv_R = \frac{s_R}{\bar{\bar{Y}}}$	coefficient of variation between laboratories (8)

Table 7: Literature data [28, 26, 27] for repeatability and reproducibility in mechanical testing.

Source	Material	T °C	*YS* MPa	cv_r	cv_R
[26]	EC-H19 aluminum	20.0	158.4	0.0210	0.0210
[26]	2024-T351 aluminum	20.0	362.9	0.0140	0.0150
[26]	AISI A105 steel	20.0	402.4	0.0140	0.0250
[26]	SS316 stainless steel	20.0	480.1	0.0140	0.0410
[26]	Inconel 600	20.0	268.3	0.0090	0.0220
[26]	SAE 51410 steel	20.0	967.5	0.0090	0.0160
[27]	AA5754 Al	20.0	105.7	NA	0.0160
[27]	AA51802-O	20.0	126.4	NA	0.0100
[27]	AA6016-T4	20.0	127.2	NA	0.0110
[27]	DX56, low-carbon steel	20.0	162.0	NA	0.0230
[27]	HR3 steel plate	20.0	228.6	NA	0.0410
[27]	ZStE 180 steel	20.0	267.1	NA	0.0500
[27]	S355 steel plate	20.0	427.6	NA	0.0310
[27]	SS316L stainless steel	20.0	230.7	NA	0.0350
[27]	X2CrNi18-10 stainless steel	20.0	303.8	NA	0.0330
[27]	X2CrNiMo18-10	20.0	353.3	NA	0.0390
[27]	30NiCrMo16 high strength steel	20.0	1039.9	NA	0.0100
[27]	Nimonic 75 CRM 661	20.0	302.1	NA	0.0180
[27]	Nimonic 75 CRM 661	20.0	298.1	NA	0.0200
[28]	SS304 stainless steel	316.0	127.3	0.0580	0.1040
[28]	Low-carbon steel	316.0	236.4	0.0110	0.0350
[28]	2.25 Cr 1Mo steel	316.0	454.7	0.0230	0.0330
[28]	A286 stainless steel	316.0	699.5	0.0280	0.0300
[28]	SS304 stainless steel	593.0	101.4	0.0770	0.0990
[28]	Low-carbon steel	593.0	133.0	0.0390	0.1040
[28]	2.2.5Cr 1Mo ferritic steel	593.0	337.9	0.0260	0.0670
[28]	A286 stainless steel	593.0	642.2	0.0260	0.0350
This study	Al 2024-T351	20.0	346.2	0.0111	0.0197

Notes:
YS= *YS*(0.2 % offset)
NA = data not available

Table 8: Analysis of variance tables for assessing the effect of lubrication on modulus and yield strength.

```
% Table generated on 2010-09-02 19:36:09
Analysis of Variance Table

Model 1: E ~ 1
Model 2: E ~ Lubricated
  Res.Df     RSS Df Sum of Sq      F  Pr(>F)
1     52 1180.16
2     51  971.31  1    208.85 10.966 0.00171 **
---
Signif. codes:  0 *** 0.001 ** 0.01 * 0.05 . 0.1   1

Analysis of Variance Table

Model 1: YS02 ~ 1
Model 2: YS02 ~ Lubricated
  Res.Df    RSS Df Sum of Sq      F    Pr(>F)
1     52 2655.5
2     51 1223.9  1    1431.6 59.653 3.939e-10 ***
---
Signif. codes:  0 *** 0.001 ** 0.01 * 0.05 . 0.1   1
```

Key to parameters in table:
`E`: E, elastic modulus
`YS02`: *YS*(0.2 % offset), 0.2 % offset yield strength
Data from laboratories that did not report the lubrication are omitted. Model 1 is the mean value of of the parameter, and model 2 computes the mean value for the lubricated ("Yes") and unlubricated ("No") cases.