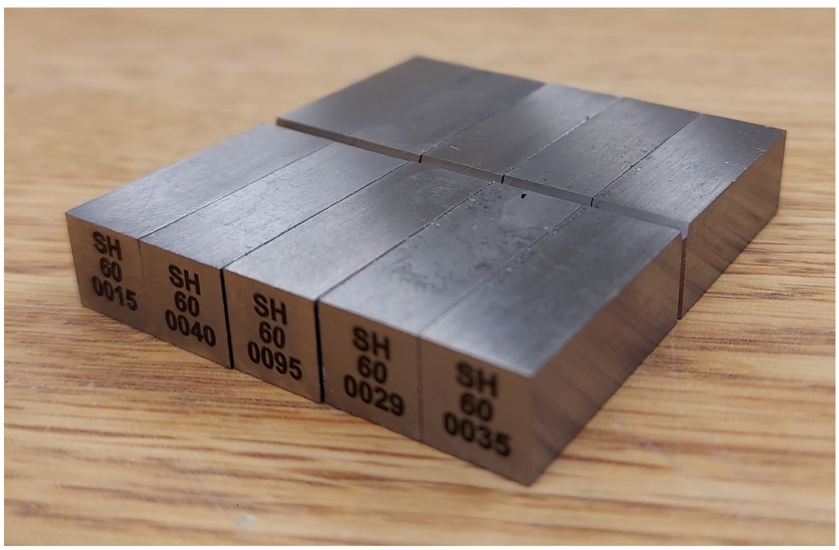
NISTIR 8424

Influence of Side-Groove Depth and Heat Treatment on Super-High Energy Charpy Specimens of 9310 Steel



Enrico Lucon

This publication is available free of charge from:
https://doi.org/10.6028/NIST.IR.8424

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

NISTIR 8424

Influence of Side-Groove Depth and Heat Treatment on Super-High Energy Charpy Specimens of 9310 Steel

Enrico Lucon
Applied Chemicals and Materials Division
Material Measurement Laboratory

This publication is available free of charge from:
https://doi.org/10.6028/NIST.IR.8424

June 2022

U.S. Department of Commerce
Gina M. Raimondo, Secretary

National Institute of Standards and Technology
Laurie E. Locascio, NIST Director and Under Secretary of Commerce for Standards and Technology

National Institute of Standards and Technology Internal Report 8424
Natl. Inst. Stand. Technol. Interag. Intern. Rep. 8424, 19 pages (June 2022)

This publication is available free of charge from:
https://doi.org/10.6028/NIST.IR.8424

Abstract

NIST provides certified reference Charpy specimens to companies and laboratories around the world for the indirect verification of impact machines in accordance with ASTM E23. Specimens are available at three energy levels, low (~15 J at -40 °C), high (~100 J at -40 °C), and super-high (~200 J at room temperature). Specimens of this latter energy level have been out of stock from the late 2000s until the late 2010s, when specimens of AISI 9310 steel were found to be suitable for standardization, provided they were side-grooved in order to prevent the formation of shear lips. This study was aimed at establishing an optimal combination of heat treatment (specifically, final tempering temperature) and side-groove depth for Charpy specimens of 9310 steel, and was conducted by testing almost 900 specimens corresponding to six different combinations. As a result of this study, a recommendation for the heat treatment and side-groove depth of future super-high energy specimen lots was formulated.

Key words

ASTM E23; final tempering temperature; NIST Charpy Machine Program; shear lips formation; side-groove depth; super-high energy Charpy specimens.

Table of Contents

Abstract **1**
Key words **1**
1. Introduction **4**
2. Development of Super-High Energy Charpy Specimens of 9310 Steel (2010-2018) . **5**
3. Conditions Investigated and Test Matrix **10**
4. Test Results **11**
4.1. Heat Treatment A – Charpy Tests 11
4.2. Heat Treatment B – Charpy Tests 11
4.3. Heat Treatment C – Charpy Tests 12
4.4. Hardness Measurements 13
5. Discussion **13**
6. Conclusions **15**
References **16**

List of Tables

Table 1 - Results of the Charpy tests performed in January 2017 on lot 3Ni1 (9310 steel). N is the number of tests and KV is the mean absorbed energy........ 6
Table 2 – Additional results of the Charpy tests performed in January 2017 on lot 3Ni1 (9310 steel). 6
Table 3 - Results of the Charpy tests performed in June 2017 on lot 3Ni2 (first series).......... 7
Table 4 - Results of the Charpy tests performed in July 2017 on lot 3Ni2 (second series)...... 7
Table 5 - Results of the Charpy tests performed in February 2018 on side-grooved specimens from lot 3Ni2........ 8
Table 6 - Results of the Charpy tests performed in September 2018 on 10 % side-grooved specimens from lot 3Ni2. 8
Table 7 - Results of the Charpy tests performed in December 2018 on the first SH-51 lot of 10 % side-grooved specimens........ 9
Table 8 - Results of the Charpy tests performed in February 2019 on the second SH-51 lot of 10 % side-grooved specimens........ 9
Table 9 - Results of the Charpy tests performed in April 2019 on the third SH-51 lot of 10 % side-grooved specimens. 9
Table 10 - Timeline of the development of super-high energy Charpy specimens from 9310 steel. 9
Table 11 - Test matrix for this investigation........ 10
Table 12 - Results of Charpy tests for the different groups of heat treatment A (SD = standard deviation; SE = standard error; KV_{min}, KV_{max} = minimum and maximum value of absorbed energy). 11
Table 13 – Overall Charpy test results for heat treatment A (U = expanded uncertainty). 11

Table 14 - Results of Charpy tests for the different groups of heat treatment B. 12
Table 15 – Overall Charpy test results for heat treatment B... 12
Table 16 - Results of Charpy tests for the different groups of heat treatment C. 13
Table 17 – Overall Charpy test results for heat treatment C... 13
Table 18 - Hardness measurement results. 13

List of Figures

Figure 1 - Examples of asymmetrical (left) and symmetrical (right) fracture for Charpy specimens. 4
Figure 2 - Absorbed energy from 9310 steel Charpy specimens with different heat treatments. 5
Figure 3 - Fracture surfaces of a 10 % side-grooved specimen of 9310 steel, showing the absence of shear lips. 8
Figure 4 - Unbroken super-high energy specimen... 12
Figure 5 - Effect of heat treatment and side-groove depth on absorbed energy. 14
Figure 6 - Effect of heat treatment and side-groove depth on sample size. 14
Figure 7 - Effect of heat treatment on Rockwell C hardness. 15

1. Introduction

Since 1989, the NIST Charpy Machine Verification Program in Boulder, Colorado [1], has supplied certified reference specimens to thousands of companies and laboratories around the world, to be used for the indirect verification of machines according to ASTM E23 [2].

NIST offers indirect verification Charpy specimens at three absorbed energy levels: low (13 J to 20 J at -40 °C), high (88 J to 136 J at -40 °C), and super-high (176 J to 244 J at room temperature). These latter specimens were introduced in the late 1990s, to address the increase in toughness and ductility of modern steels. For about 10 years, super-high energy specimens were made of T-200 (18Ni) maraging steel, but in the late 2000s it became increasingly difficult, and eventually impossible, for NIST to procure T-200 steel with sufficient quality and homogeneity. As a consequence, super-high energy specimen went out of stock around 2008.

Starting in 2010, it was decided to investigate an alternative steel for the production of super-high energy specimens: AISI 9310 steel, which had been used for several years by the European Union to prepare samples with 150 J nominal absorbed energy.

Early investigations showed a statistically significant influence of fracture mode (symmetrical vs. asymmetrical, Figure 1) on the energy absorbed by 9310 steel Charpy specimens [3]. Specifically, when both shear lips formed during the fracture process were located on the same broken specimen half (symmetrical fracture), absorbed energy tended to be lower than when each half featured one shear lip only (asymmetrical fracture). The difference, as can be seen in Section 2, was of the order of 15 J – 20 J.

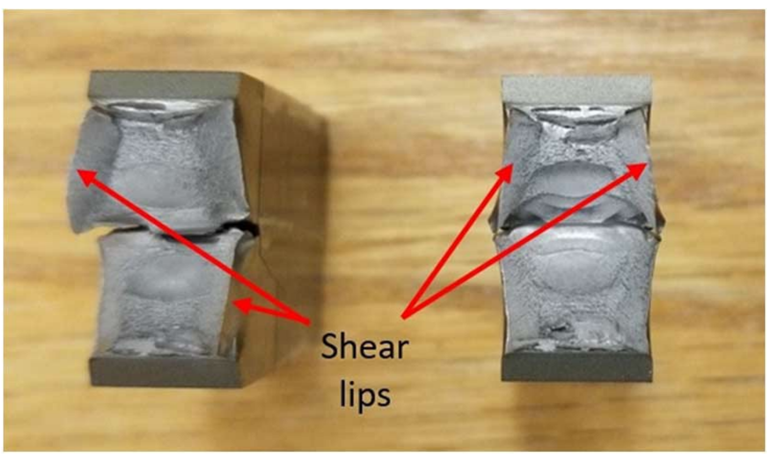


Figure 1 - Examples of asymmetrical (left) and symmetrical (right) fracture for Charpy specimens.

In the presence of shear lips on the broken specimen, it was practically impossible to fulfil the NIST requirement on the variability of certified Charpy specimens (sample size[1] calculated from testing 75 specimens on the three NIST reference machines < 5.0) [1], as test results exhibited a bimodal distribution between symmetrical and asymmetrical fractures,

[1] The sample size for NIST lots is given by:

$$n_{SS} = \left(\frac{3s_p}{E}\right)^2 ,$$

where s_p = pooled standard deviation of the three NIST reference machines and E = larger between 1.4 J and 5 % of the mean absorbed energy. It corresponds to the minimum number of specimens a customer must test for the results to be statistically comparable with the NIST certified absorbed energy.

with the latter fractures absorbing significantly more energy than the former. In order to prevent shear lips formation, it was decided to side-groove the specimens of 9310 steel.

The experimental activities performed on 9310 steel in the period 2010-2018, which eventually resulted in the reinstatement of super-high energy Charpy specimens in the NIST catalog, are summarized below.

2. Development of Super-High Energy Charpy Specimens of 9310 Steel (2010-2018)

The earliest activities on 9310 steel as a replacement of T-200 for super-high energy Charpy specimens date back to 2010-2011, when several preliminary batches, corresponding to slightly different heat treatments, were tested on the NIST reference Charpy machines.

For these early batches, the basic heat treatment consisted of:

- normalization, followed by air cool,
- hardening, followed by oil quench,
- first temper, followed by air cool, and
- final temper, followed by air cool.

The variables investigated were temperature and duration of the hardening step, temperature of the first temper, and temperature of the second temper. The results of the Charpy tests on 8 different material conditions are presented in Figure 2 as a function of tempering temperature (1st temper). In Figure 2, the typical energy range mentioned in ASTM E23 for super-high energy specimens (176 J to 244 J) is indicated by dashed horizontal lines.

The batches that exhibited the lowest coefficients of variation (CV = standard deviation/average between 2.2 % and 4.2 %) yielded absorbed energies that were too high (above 250 J). The best compromise between mean energy (204.5 J) and CV (4.9 %) corresponds to specimens tempered at 149 °C and re-tempered at 482 °C for 2 h.

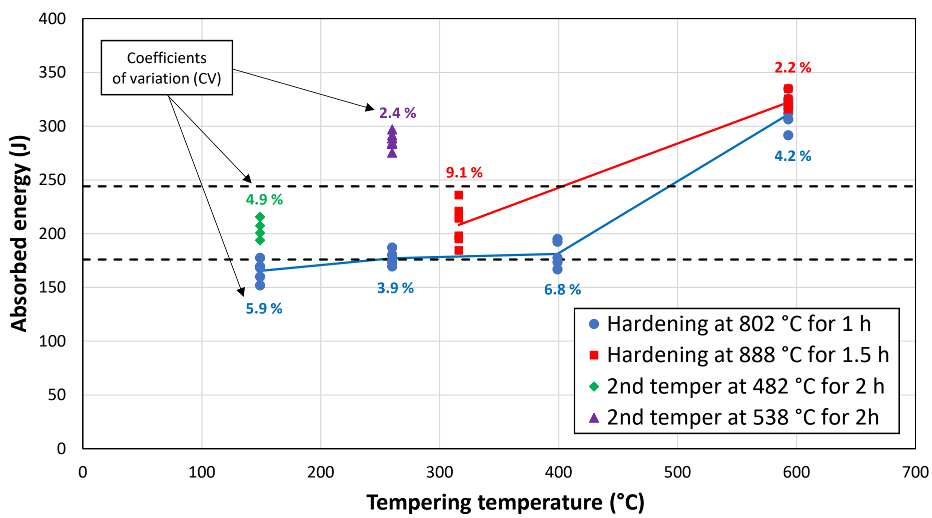


Figure 2 - Absorbed energy from 9310 steel Charpy specimens with different heat treatments.

After a few years when activities were put on hold, in 2017 work resumed on a more complex heat treatment, consisting of:

- normalization;
- temperature increase and stay;
- furnace cooling, followed by gas cooling;
- neutral hardening, followed by agitated oil quench, air cool, and wash;
- first temper;
- cryo-treatment/deep freeze;
- final temper.

The first lot of 9310 steel specimens, called 3Ni1, was tested in January 2017 (75 tests, 25 on each NIST reference machine). Test results highlighted the already mentioned bimodal behavior of the steel, caused by the development of shear lips and the ensuing asymmetrical/ symmetrical fracture.

Table 1 presents a summary of Charpy test results from lot 3Ni1. Approximately one third of the specimens broke asymmetrically, and their average absorbed energy was almost 15 J higher than for symmetrical fractures. Statistically, the two groups of specimens were found to be extremely different based on a two-sample *t*-test, which returned a two-tail probability value[2] $p = 2.36 \times 10^{-29}$. Table 1 also shows that, while the overall group of specimens tested yielded a clearly unacceptable sample size of 15.5, asymmetrical and symmetrical fractures, separately considered, exhibited acceptable variability (n_{SS} = 2.3 and 2.0, respectively).

Table 1 - Results of the Charpy tests performed in January 2017 on lot 3Ni1 (9310 steel). *N* is the number of tests and $\overline{KV}$ is the mean absorbed energy.

Tests	***N* (%)**	**$\overline{KV}$ (J)**	**CV (%)**	**n_{SS}**
All	75 (100 %)	199.34	6.5	**15.5**
Asymmetrical	28 (37 %)	214.77	2.7	**2.3**
Symmetrical	47 (63 %)	190.47	2.7	**2.0**

Another feature of interest that emerged from the January 2017 tests was the different behavior exhibited by broken and unbroken[3] specimens. At energy levels above 200 J, it is common to have specimens plastically deformed during impact and pushed through the anvils by the swinging hammer before complete fracture. For the tests on 3Ni1, the statistics for broken and unbroken specimens are summarized in Table 2.

Table 2 – Additional results of the Charpy tests performed in January 2017 on lot 3Ni1 (9310 steel).

Tests	**N (%)**	**$\overline{KV}$ (J)**	**CV (%)**	**n_{SS}**
All	75 (100 %)	199.34	6.5	**15.5**
Broken	65 (87 %)	197.05	6.2	**13.6**
Unbroken	10 (13 %)	213.98	2.4	**2.1**

[2] In a *t*-test, the mean values of two groups are considered statistically different if $p < 0.05$, where $\alpha = 0.05$ is the typical confidence level.
[3] If the tested specimen is still in one piece, but can be easily broken by pushing the two hinged halves together and then pulling them apart with the bare hand (without using tools), the specimen is defined "finger broken" and can be considered equivalent to a fully broken specimen.

Most 3Ni1 tested specimens (87 %) were broken or finger broken, and their mean absorbed energy resulted in significantly lower values ($p = 3.41 \times 10^{-8}$ from a two-sample t-test) than for unbroken specimens. These latter also yielded an acceptable variability (n_{SS} = 2.1), unlike broken specimens (n_{SS} = 13.6).[4]

The main lessons learned from testing the 3Ni1 lot were:

- the formation of shear lips, with the ensuing dichotomy between asymmetrical and symmetrical fractures, should be avoided;
- comparable numbers of broken or unbroken specimens are also not desirable.

A modified heat treatment was applied to a second lot (3Ni2) of 9310 steel specimens in February 2017. The temperatures of the increase after normalization, the neutral hardening, and the cryo-treatment were changed, as well as the duration of the neutral hardening. This lot was characterized in June-July 2017.

The statistics from the first test series (June 2017) and the second test series (July 2017) are provided in Table 3 and Table 4, respectively.

Table 3 - Results of the Charpy tests performed in June 2017 on lot 3Ni2 (first series).

Tests	***N* (%)**	**$\overline{KV}$ (J)**	**CV (%)**	**n_{SS}**
All	75 (100 %)	190.20	6.0	**12.6**
Asymmetrical	19 (25 %)	207.58	3.7	**5.3**
Symmetrical	56 (75 %)	184.30	2.1	**1.1**
Broken	62 (83 %)	186.29	4.2	**6.6**
Unbroken	13 (17 %)	208.83	2.7	**2.1**

Table 4 - Results of the Charpy tests performed in July 2017 on lot 3Ni2 (second series).

Tests	***N* (%)**	**$\overline{KV}$ (J)**	**CV (%)**	**n_{SS}**
All	98 (100 %)	192.73	5.9	**12.7**
Asymmetrical	26 (27 %)	209.60	3.0	**3.1**
Symmetrical	72 (73 %)	186.64	2.4	**2.1**
Broken	92 (94 %)	191.77	5.7	**11.8**
Unbroken	6 (%)	207.55	3.9	**6.5**

The results from 3Ni2 substantially confirmed the trends observed for 3Ni1. Overall, the modified heat treatment slightly increased the average absorbed energy[5], but didn't significantly change the variability. More specimens fully broke for 3Ni2 than for 3Ni1, but the bimodal nature of the steel caused by the shear lips remained.

The remaining specimens from 3Ni2 were used in 2018 to investigate side-grooving, and particularly the effectiveness in preventing the formation of shear lips. Two side-groove depths, *SGD*, were investigated: 5 % of the total thickness (0.25 mm per side – 45 specimens) and 10 % (0.5 mm/side – 45 specimens). The results of these Charpy tests are

[4] Interestingly, fully broken specimens (45 % of the tests performed) provided an acceptable sample size (3.2), while finger-broken specimens (41 % of the tests performed) showed unacceptable scatter (n_{SS} = 11.9), When combined, fully broken and finger broken samples caused the sample size to be unacceptable (13.6).

[5] The two lots were found not statistically different, based on a t-test ($p = 0.15 > 0.05$).

summarized in Table 5. Doubling the depth of the side-grooves caused a 13 % decrease of $\overline{KV}$ and a decrease of sample size (from 5.8, unacceptable, to 2.8, acceptable).

Table 5 - Results of the Charpy tests performed in February 2018 on side-grooved specimens from lot 3Ni2.

Specimens	***N***	**$\overline{KV}$ (J)**	**CV (%)**	**n_{SS}**
5 % side-grooved	45	197.86	3.9	**5.8**
10 % side-grooved	45	171.84	4.3	**2.8**

Seventy-five additional 10 % side-grooved specimens from lot 3Ni2 were tested in September 2018. The results obtained (Table 6) confirmed both the energy level and the sample size.

Table 6 - Results of the Charpy tests performed in September 2018 on 10 % side-grooved specimens from lot 3Ni2.

Specimens	***N***	**$\overline{KV}$ (J)**	**CV (%)**	**n_{SS}**
10 % side-grooved	75	165.27	2.7	**2.7**

As seen in Figure 3, side-grooving specimens by 0.5 mm/side was fully successful in preventing the formation of shear lips. However, the average absorbed energies for 10 % side-grooved specimens was below the recommended lower limit in ASTM E23-18 (176 J). Nevertheless, this specimen lot was considered useable by NIST and re-labeled SH-50.

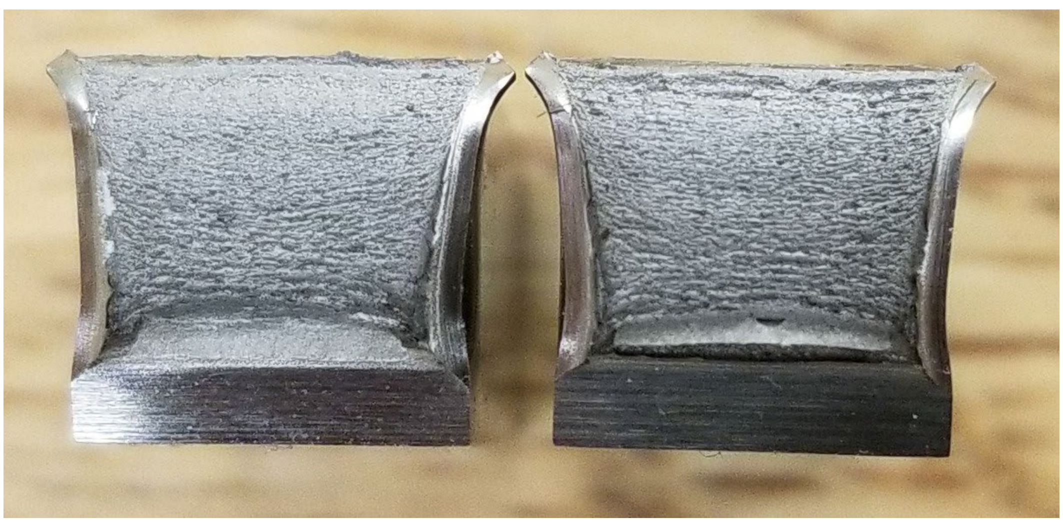


Figure 3 - Fracture surfaces of a 10 % side-grooved specimen of 9310 steel, showing the absence of shear lips.

In December 2018, a new super-high energy lot (labeled SH-51) was prepared after tweaking the heat treatment, in order to increase the mean absorbed energy (specifically, by increasing the temperature of the final temper). The tests performed (Table 7) showed a clear increase of $\overline{KV}$, but also yielded an unacceptable sample size (6.4). Out of 75 specimens tested, the vast majority (54, or 72 %) did not break. Of the remaining 21 samples, 10 (13 %) fully broke and 11 (15 %) were finger-broken.

Table 7 - Results of the Charpy tests performed in December 2018 on the first SH-51 lot of 10 % side-grooved specimens.

Specimens	N	$\overline{KV}$ (J)	CV (%)	n_{SS}
10 % side-grooved	75	211.81	4.2	6.4

A second lot of 75 SH-51 specimens was tested in February 2019, after increasing again the temperature of the final temper. The results (Table 8) showed a further increase of $\overline{KV}$ and an acceptable sample size of 2.5.

Table 8 - Results of the Charpy tests performed in February 2019 on the second SH-51 lot of 10 % side-grooved specimens.

Specimens	N	$\overline{KV}$ (J)	CV (%)	n_{SS}
10 % side-grooved	75	234.11	2.7	2.5

The results of the tests performed in April 2019 on a third SH-51 lot substantially confirmed the absorbed energy level and the sample size (Table 9).

Table 9 - Results of the Charpy tests performed in April 2019 on the third SH-51 lot of 10 % side-grooved specimens.

Specimens	N	$\overline{KV}$ (J)	CV (%)	n_{SS}
10 % side-grooved	75	227.26	3.5	4.2

At the end of the development process described above and summarized in the timeline of Table 10, super-high energy Charpy specimens were reinstated in the NIST Standard Reference Material (SRM) catalog (lots SH-50 and SH-51, both 10 % side-grooved).

Table 10 - Timeline of the development of super-high energy Charpy specimens from 9310 steel.

<table>
<tr><th>Date</th><th>Lot Id</th><th>Side-grooving</th><th>$\overline{KV}$ (J)</th><th>n_{SS}</th></tr>
<tr><td>1/2017</td><td>3Ni1</td><td>0 %</td><td>199.34</td><td>15.5</td></tr>
<tr><td>6/2017</td><td rowspan="3">3Ni2</td><td rowspan="2">0 %</td><td>190.20</td><td>12.6</td></tr>
<tr><td>7/2017</td><td>192.73</td><td>12.7</td></tr>
<tr><td rowspan="2">2/2018</td><td>5 %</td><td>197.86</td><td>5.8</td></tr>
<tr><td rowspan="2">SH-50</td><td rowspan="2">10 %</td><td>171.84</td><td>2.8</td></tr>
<tr><td>9/2018</td><td>165.27</td><td>2.7</td></tr>
<tr><td>12/2018</td><td rowspan="3">SH-51</td><td rowspan="3">10 %</td><td>211.81</td><td>6.4</td></tr>
<tr><td>2/2019</td><td>234.11</td><td>2.5</td></tr>
<tr><td>4/2019</td><td>227.26</td><td>4.2</td></tr>
</table>

Between 2019 and 2022, seven additional super-high energy lots were produced. As some of these lots provided unacceptable samples sizes, the depth of the side-grooves was progressively increased in order to achieve the desired variability. The side-grooving percentage increased from 10 % (0.5 mm/side) to 18 % (0.85 mm/side) in successive steps.

However, increasing *SGD* decreases the area of the ligament below the notch, and causes a reduction of the mean absorbed energy.

We therefore decided to conduct an investigation aimed at establishing the ideal combination of *SGD* and $\overline{KV}$ (as determined by a suitable heat treatment, with particular regard to the temperature of the final temper). The results and conclusions of this investigation are provided in the remainder of this report.

3. Conditions Investigated and Test Matrix

Three heat treatments (A, B, and C) and two side-groove depths (0.9 mm/side and 1 mm/side) were investigated, for a total of six conditions. The three heat treatments only differed in the final tempering temperature: the temperature for heat treatment B was 14 °C (25 °F) higher than A, and the final tempering temperature of C was 14 °C higher than B.

The maximum side-groove depth considered was 1 mm/side, for a total thickness reduction of 20 %. This is the typical depth recommended for fracture toughness specimens by ASTM Test Methods such as E1820 and E1921. Deeper side-grooves were expected to cause an excessive reduction of the mean absorbed energy.

For each condition, a number of specimens equivalent to an ordinary NIST indirect verification specimen lot (150, split between 75 in the first lot and 75 in the second lot)[6] were tested at room temperature (21 °C ± 1 °C) on the three NIST reference machines. For each condition, 50 specimens were tested on each reference machine. For some conditions, less than 150 specimens were actually available, due to some samples being unusable after machining.

In order to simulate the certification process of an ordinary NIST indirect verification lot, for each condition the first 75 specimens and the remaining 75 specimens were tested on different days. The overall test matrix is shown in Table 11.

Table 11 - Test matrix for this investigation.

Heat Treatment	***SGD* (mm/side)**	***N***
A	0.9	150
	1	149
B	0.9	148
	1	145
C	0.9	148
	1	150
Total tests performed:		890

[6] During the NIST certification process, the first group of 75 specimens is called "pilot" lot, and the second group of specimens is called "production" lot.

Before Charpy testing, Rockwell C hardness (HRC) measurements were also performed on 30 randomly selected Charpy specimens, 10 for each of the heat treatments (obviously, hardness does not depend on side-groove depth).

4. Test Results

4.1. Heat Treatment A – Charpy Tests

Heat treatment A corresponds to the lowest final tempering temperature. 150 specimens were tested with *SGD* = 0.9 mm/side (18 % thickness reduction) and 149 specimens with *SGD* = 1 mm/side (20 % thickness reduction). All tests were performed at room temperature (21 °C ± 1 °C), one third on each reference machine. For each side-groove depth, two groups of tests (group 1 and group 2) were performed on different days under identical experimental conditions.

Charpy test results for heat treatment A are summarized in Table 12 (individual groups) and Table 13 (reference absorbed energies, expanded uncertainties, and sample sizes).

Table 12 - Results of Charpy tests for the different groups of heat treatment A (SD = standard deviation; SE = standard error; KV_{min}, KV_{max} = minimum and maximum value of absorbed energy).

SGD (mm/side)	Group	No. of tests	$\overline{KV}$ (J)	SD (J)	SE (J)	KV_{min} (J)	KV_{max} (J)	CV (%)	n_{SS}
0.9	1	75	179.33	4.49	0.52	169.65	190.26	2.5	**2.103**
	2	75	178.60	4.75	0.55	169.93	190.05	2.7	**2.092**
1	1	75	166.15	3.59	0.42	159.36	176.89	2.2	**1.103**
	2	74	166.66	4.14	0.48	158.29	176.16	2.5	**1.592**

Table 13 – Overall Charpy test results for heat treatment A (U = expanded uncertainty).

SGD (mm/side)	No. of tests	$\overline{KV}$ (J)	*U* (J)	n_{SS}
0.9	150	178.96	0.70	**2.103**
1	149	166.43	0.52	**1.348**

None of the 299 Charpy specimens tested fully broke or could be broken by finger force. For all specimens tested, the two halves remained connected by a thin ligament less than 1 mm thick (Figure 4), with a plastic bending angle of approximately 50°-55° that allowed the unbroken specimen to be pushed through the anvils by the swinging hammer.

4.2. Heat Treatment B – Charpy Tests

Heat treatment B corresponds to a final tempering temperature 14 °C (25 °F) higher than heat treatment A. 148 specimens were tested with *SGD* = 0.9 mm/side and 145 specimens with *SGD* = 1 mm/side. All tests were performed at room temperature (21 °C ± 1 °C) on the three NIST reference machines. For each side-groove depth, two groups of tests (group 1 and group 2) were performed on different days under identical experimental conditions.

Charpy test results for heat treatment B are summarized in Table 14 (individual groups) and Table 15 (reference absorbed energies, expanded uncertainties, and sample sizes).

Table 14 - Results of Charpy tests for the different groups of heat treatment B.

***SGD* (mm/side)**	**Group**	**No. of tests**	$\overline{KV}$ **(J)**	**SD (J)**	**SE (J)**	KV_{min} **(J)**	KV_{max} **(J)**	**CV (%)**	n_{SS}
0.9	1	74	186.74	4.37	0.51	180.74	198.04	2.3	**1.382**
	2	74	187.39	5.03	0.59	177.62	200.86	2.7	**2.374**
1	1	73	166.21	2.31	0.27	161.12	176.13	1.4	**0.576**
	2	72	166.62	2.59	0.31	160.37	172.78	1.6	**0.764**

Table 15 – Overall Charpy test results for heat treatment B.

***SGD* (mm/side)**	**No. of tests**	$\overline{KV}$ **(J)**	***U* (J)**	n_{SS}
0.9	148	187.07	0.694	**1.879**
1	145	166.42	0.374	**0.670**

None of the 293 Charpy tested specimens fully broke or could be broken by finger force.

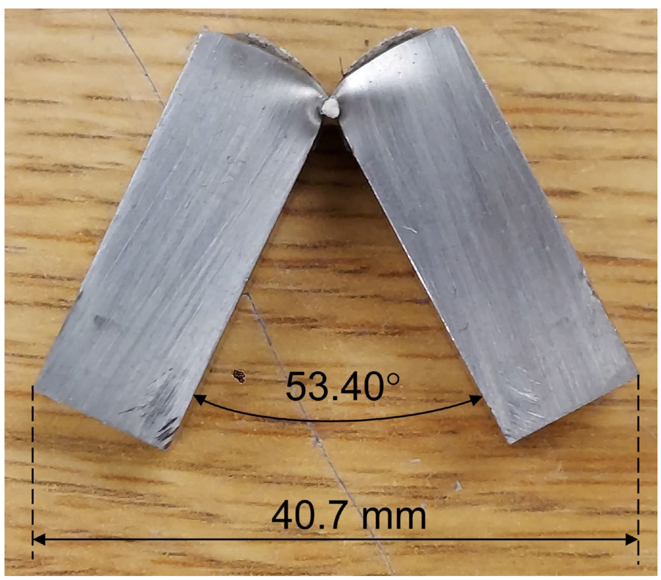


Figure 4 - Unbroken 18 % side-grooved super-high energy specimen.

4.3. Heat Treatment C – Charpy Tests

Heat treatment C corresponds to the highest final tempering temperature, 14 °C (25 °F) higher than heat treatment B. 148 specimens with *SGD* = 0.9 mm/side and 150 specimens with *SGD* = 1 mm/side were tested. All tests were performed at room temperature (21 °C ± 1 °C) on the three NIST reference machines. For each side-groove depth, two groups of tests (group 1 and group 2) were performed on different days under identical experimental conditions.

Charpy results for heat treatment C are summarized in Table 14 (individual groups) and Table 15 (reference absorbed energies, expanded uncertainties, and sample sizes).

Table 16 - Results of Charpy tests for the different groups of heat treatment C.

***SGD* (mm/side)**	**Group**	**No. of tests**	**$\overline{KV}$ (J)**	**SD (J)**	**SE (J)**	**KV_{min} (J)**	**KV_{max} (J)**	**CV (%)**	**n_{SS}**
0.9	1	75	190.09	3.96	0.46	181.15	200.49	2.1	**1.421**
	2	73	188.71	4.90	0.57	179.13	205.30	2.6	**1.956**
1	1	75	186.25	5.34	0.62	175.11	199.72	2.9	**3.015**
	2	75	185.93	5.32	0.61	173.73	200.17	2.9	**2.977**

Table 17 – Overall Charpy test results for heat treatment C.

***SGD* (mm/side)**	**No. of tests**	**$\overline{KV}$ (J)**	***U* (J)**	**n_{SS}**
0.9	148	189.39	1.022	**1.956**
1	150	186.09	0.867	**2.996**

None of the 298 tested specimens fully broke or could be broken by finger force.

4.4. Hardness Measurements

The results of Rockwell C (HRC) measurements on 30 randomly selected specimens (10 for each heat treatment) are summarized in Table 18. We note a decrease of hardness and an increase in measurement scatter as the final tempering temperature increases.

Table 18 - Hardness measurement results.

Heat treatment	**No. of specimens**	**$\overline{HRC}$**	**SD**	**SE**	**HRC_{min}**	**HRC_{max}**	**CV (%)**
A	10	25.30	0.15	0.05	25.10	25.45	0.59
B	10	23.79	0.26	0.08	23.35	24.20	1.08
C	10	23.08	0.52	0.16	22.15	23.80	2.25

5. Discussion

Increasing the final tempering temperature caused an increase of absorbed energy (Figure 5) and a decrease of hardness (Figure 6). In terms of material variability, a general decrease of sample size was observed with increasing final tempering temperature (Figure 7). These trends were found to be consistent for 5 of the 6 investigated conditions, however a somewhat different behavior was observed for heat treatment C and *SGD* = 1 mm: for this condition, absorbed energy remains constant between A and B, but a sharp increase is observed for heat treatment C. For this latter heat treatment, the sample size calculated for *SGD* = 1 mm is the highest among all examined conditions (close to 3).

The consequence of increasing side-groove depth is a decrease of both absorbed energy and sample size (with the exception of heat treatment C/*SGD* = 1 mm).

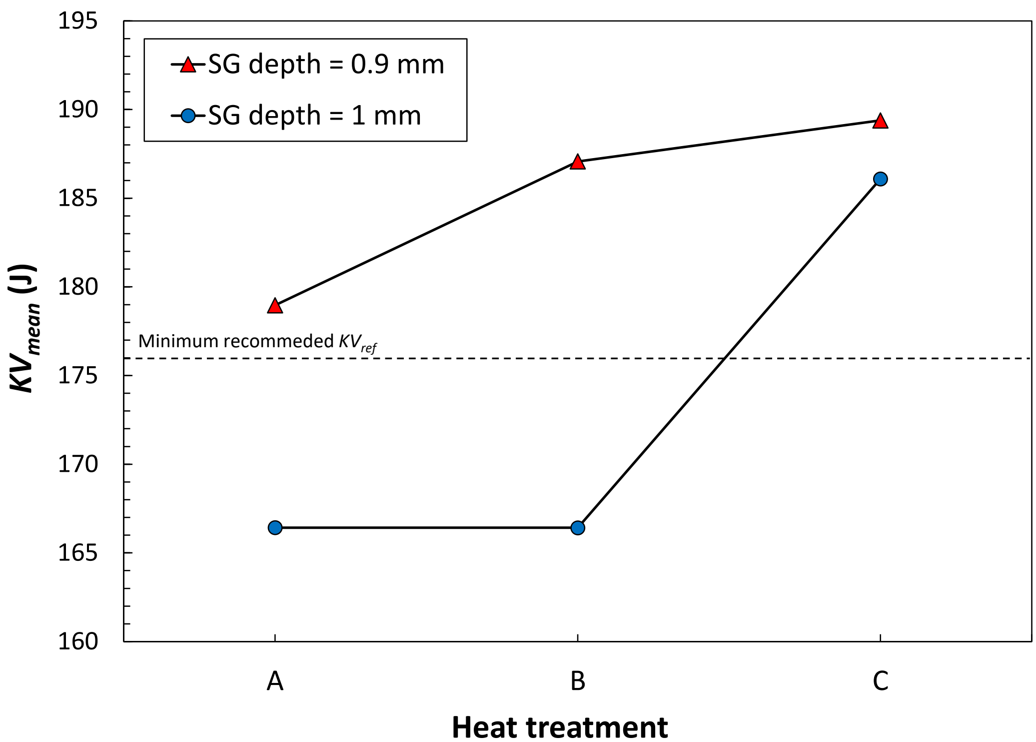


Figure 5 - Effect of heat treatment and side-groove depth on absorbed energy.

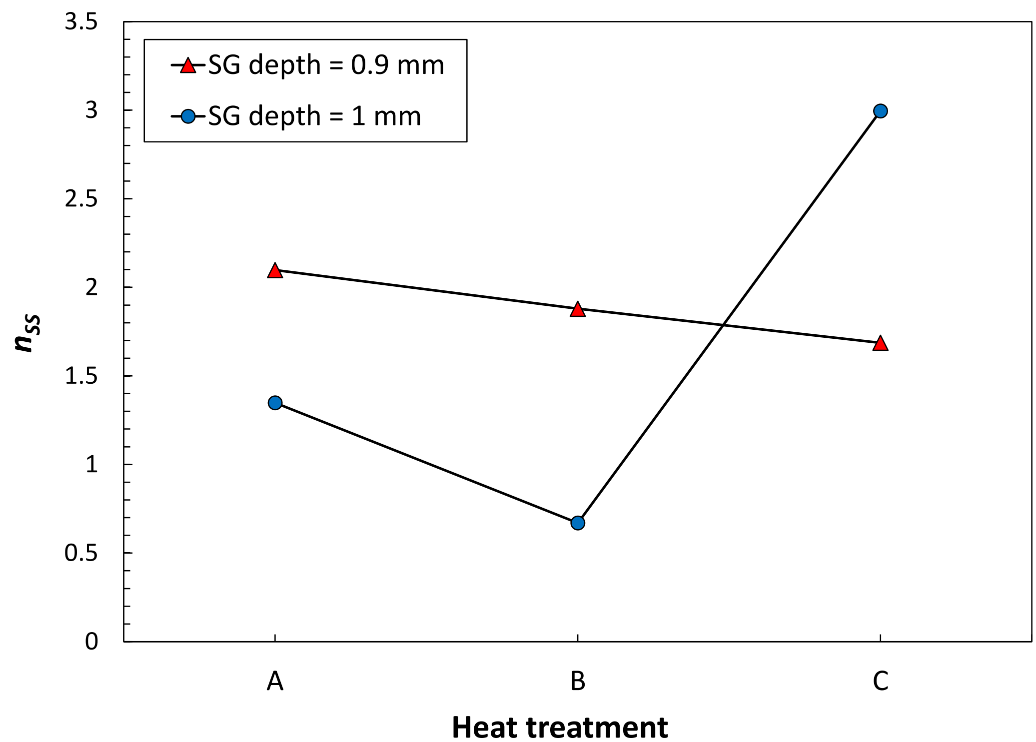


Figure 6 - Effect of heat treatment and side-groove depth on sample size.

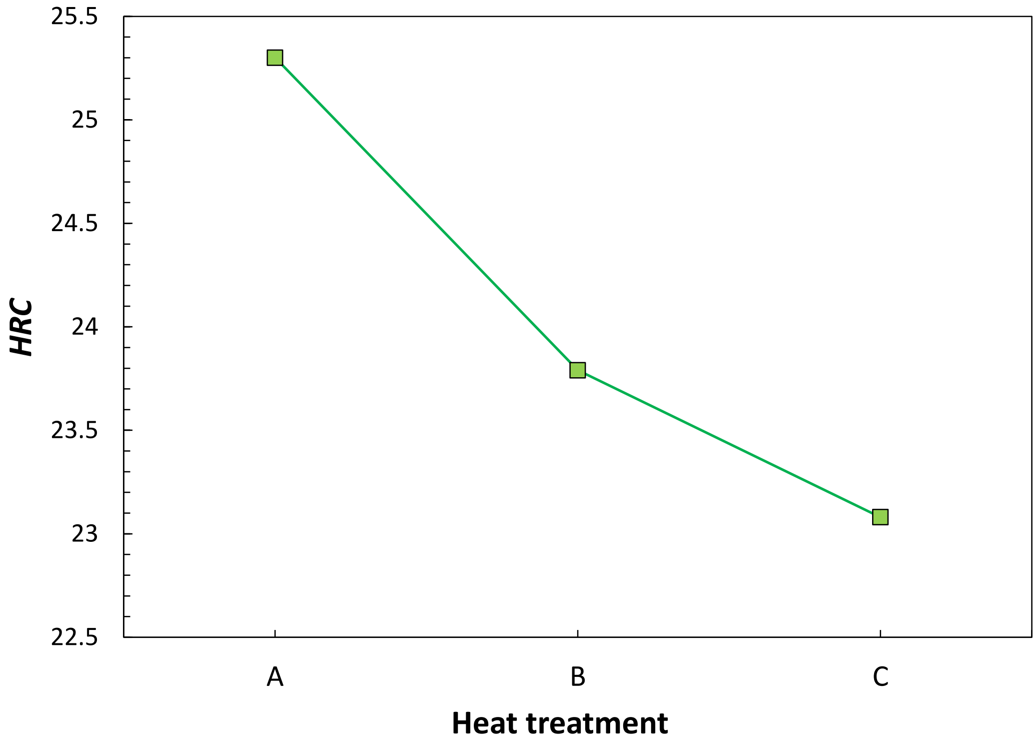


Figure 7 - Effect of heat treatment on Rockwell C hardness.

The mean values of absorbed energy are within the desired/recommended range for super-high energy specimens (176 J – 244 J) for *SGD* = 0.9 mm and for heat treatment C and *SGD* = 1 mm (Figure 5). For heat treatments A and B and *SGD* =1 mm, KV_{mean} < 176 J.

As previously mentioned, none of the 890 tested specimens exited the machine completely broken, or could be separated by pushing the hinged halves together once and then pulling them apart by mere hand force. Most likely, this is a result of the high ductility induced by the heat treatments, in that specimens plastically bend to an angle such that the sample can exit the machine through the anvils before fracturing completely.

6. Conclusions

An investigation was conducted aimed at establishing the optimal combination of heat treatment (specifically, final tempering temperature) and side-groove depth for super-high energy Charpy specimens made of 9310 steel for the NIST Charpy Machine Verification Program.

Six combinations of heat treatment and side-groove depth were investigated, by testing a total of 890 specimens on the three NIST Charpy reference machines in Boulder, Colorado. All combinations provided acceptable results according to NIST specifications (sample size < 5.0). All tested specimens remained unbroken, due to the high ductility of the steel.

The lowest variability (sample size) was observed for heat treatment B (intermediate final tempering temperature) and 20 % side-grooving (1 mm/side), but the mean absorbed energy (166 J) is lower than the recommended minimum (176 J).

The best combination between sample size (1.687) and mean absorbed energy (189 J) was found to correspond to the highest tempering temperature (heat treatment C) and 18 % side-grooving (0.9 mm/side). These conditions will be adopted in the NIST specifications for future super-high energy indirect verification lots.

References

[1] McCowan, C. N., Siewert, T. A., and Vigliotti, D. P., "Charpy Verification Program: Reports Covering 1989-2002," NIST Technical Note 1500-9, Materials Reliability Series, National Institute of Standards and Technology, September 2003. http://dx.doi.org/10.6028/NIST.TN.1500-9

[2] ASTM E23-18, "Standard Test Methods for Notched Bar Impact Testing of Metallic Materials," ASTM International, West Conshohocken, PA.

[3] Lucon, E., "Influence of Shear Lip Symmetry on the Fracture Behavior of Charpy Specimens," *Journal of Testing and Evaluation*, Vol. 47, No. 2, Jan 2019. https://doi.org/10.1520/JTE20180403