

**NIST Internal Report
NIST IR 8531**

**Optimized Heat Treatment for NIST
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to Be Tested at 21 °C**

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Abstract

The NIST Charpy Machine Verification Program has recently decided to change the test temperature for low-energy and high-energy certified reference specimens from -40 °C to 21 °C. Consequently, low-energy specimens currently tend to remain in the impact zone after fracture, with an increased risk of jamming or multiple secondary collisions between broken halves and various machine parts. Both occurrences artificially increase absorbed energy, and their likelihood should be minimized. In the study presented here, we investigated the effect of changing the final tempering temperature (T_{temp}) of low-energy specimens, with the aim of decreasing their room temperature impact toughness, and therefore lowering the chance of jamming/multiple collisions to acceptable levels (close to zero). It was found that decreasing T_{temp} from 371 °C/700 °F (current heat treatment) to 316 °C/600 °F approximately duplicates at 21 °C the testing conditions (absorbed energy and post-fracture kinetics) previously observed at -40 °C.

Keywords

Certified Charpy specimens; NIST Charpy Machine Verification Program; post-fracture kinetics; specimen jamming; tempering temperature.

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1. Introduction

The NIST Charpy Machine Verification Program [1] has supplied certified reference Charpy specimens to thousands of companies and laboratories worldwide since 1989, when the program was transferred from the US Army in Watertown, Massachusetts, who had started the program in the late 1960's [2], to NIST in Boulder, Colorado. These reference specimens are used for the indirect verification of Charpy machines in accordance with ASTM E23 [3] and ISO 148-2 [4].

NIST certified specimens are available at three energy levels: low (typically 13 J to 20 J), high (typically 90 J to 140 J), and super-high (typically 175 J to 240 J). These latter specimens were introduced in the 1990's, and have always been tested at room temperature (21 °C/70 °F). Low-energy and high-energy specimens, however, were tested at -40 °C (-40 °F) since their introduction.

In 2015, NIST first investigated the possibility of certifying low- and high-energy Charpy verification specimens tested at room temperature [5]. It was found that, in most cases, the variability (*i.e.*, data scatter) of Charpy specimen lots slightly decreased moving from -40 °C to 21 °C. The increase of test temperature also caused an increase in specimen toughness, corresponding to an approximate increase of 10 % in absorbed energy.

This toughness increase had no practical consequences for high-energy specimens. However, for low-energy specimens, the augmented absorbed energy caused a change in the post-fracture kinetics of the broken specimen halves: instead of being ejected backward¹ at high speed, as always happens at -40 °C, most of the tested specimens remained in the test area (between swinging hammer, supports, anvils, and shrouds if present). This increased the likelihood of a specimen half “jamming” between moving and stationary parts of the machine, or experiencing multiple collisions with the same machine parts. In both cases, the consequence was an artificial increase of absorbed energy (particularly significant in the case of jamming), on top of the actual fracture energy.

In 2022, the Charpy Program Leader and the Charpy Coordinator decided to change the test temperature of low- and high-energy reference specimens to 21 °C, satisfying the requests received from many NIST customers. At the time of writing, the only specimens available for testing at -40 °C were of the self-verification type, which did not include any NIST post-test service, such as the evaluation of broken specimen pictures or the issuance of an official Verification Letter.

The investigation described in this report was aimed at optimizing the heat treatment of low-energy specimens, so that the absorbed energy increase caused by the higher test temperature is effectively countered by a lower material toughness. The primary objective was to identify the final tempering temperature, T_{temp} , that would duplicate at 21 °C the mechanical behavior observed at -40 °C (similar absorbed energy level and post-fracture kinetics). The probability of jamming/multiple collisions could therefore be appropriately minimized.

According to a pivotal 2003 NIST publication describing the Charpy Machine Verification Program [6], the absorbed energy of 4340 steel is sensitive to the tempering temperature, as

¹ Backward = opposite the direction of pendulum swing.

shown in Fig. 1. Specifically, the energy corresponding to the low-energy level is achieved with a tempering temperature between 300 °C and 400 °C. Although the absorbed energy level appears substantially constant in this range, a slight decreasing trend with decreasing T_{temp} is seen in Fig. 1.

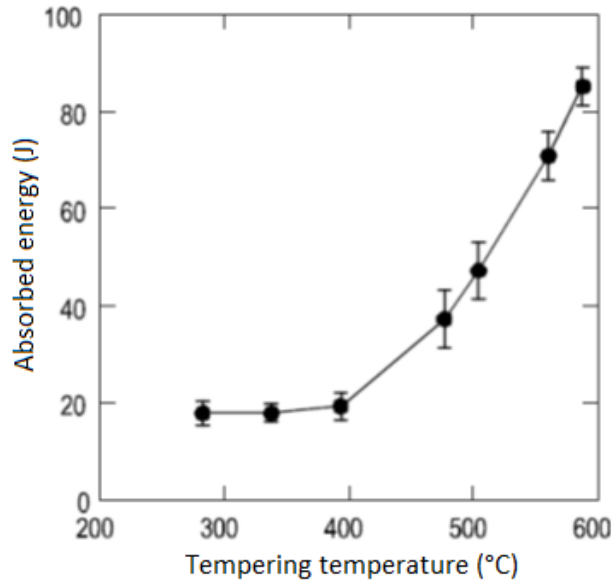


Fig. 1. Absorbed energy for 4340 steel as a function of tempering temperature, T_{temp} [6].

Within this investigation, we considered eight groups (A-H) of thirty 4340 steel specimens each, tempered for 3 hours in the range 316 °C (600 °F) to 413 °C (775 °F). For every T_{temp} , 30 Charpy specimens were heat treated, machined, and tested (10 on each of the three NIST reference machines). In the case of $T_{temp} = 371$ °C (700 °F), which is the temperature used for tempering -40 °C low-energy specimens, three groups of 30 specimens were tested:

- **Group A:** $T_{temp} = 371$ °C, $T_{test} = -40$ °C. This represents the benchmark, or the toughness condition that we aimed at reproducing when testing at 21 °C.
- **Group B:** $T_{temp} = 371$ °C, $T_{test} = 21$ °C. This group was expected to provide a higher toughness than group A, and therefore increase the likelihood of jamming/multiple collisions.
- **Group H:** $T_{temp} = 371$ °C, $T_{test} = 21$ °C. The 4340 heat from which groups A-E were obtained (heat #J3548) was used up for those first five groups, so a different steel heat (#J2803) was used for the last three groups (F, G, H). A statistical comparison between groups B and H would confirm whether the two heats could be considered equivalent for practical purposes.

Overall, 240 Charpy tests (80 on each reference machine) were performed, 30 at -40 °C and 210 at 21 °C. The complete test matrix is shown in Table 1.

Table 1. Test matrix.

Group	Heat #	T_{temp} (°C)	T_{temp} (°F)	T_{test} (°C)	<i>Specimens tested</i>
A	J3548	371	700	-40	30
B	J3548	371	700	21	30
C	J3548	385	725	21	30
D	J3548	399	750	21	30
E	J3548	413	775	21	30
F	J2280	343	650	21	30
G	J2280	316	600	21	30
H	J2280	371	700	21	30

2. Test results

The most straightforward evidence of a low-energy specimen jamming after impact is the unusually high absorbed energy value. Additionally, obvious marks are typically visible on one or both broken halves. In case of multiple post-fracture collisions, the increase in energy is less evident, and specimen marks are less pronounced.

We used a simple statistical outlier test (Grubbs' test, [7]) to identify the specimens that might have experienced jamming or multiple collisions. This was performed on each group of 10 tests conducted on an individual machine. Only high energy outliers were considered.

2.1. Group A ($T_{temp} = 371\text{ °C}$, $T_{test} = -40\text{ °C}$)

Group A represents the benchmark of the study, or the -40 °C condition that needed to be duplicated at 21 °C . The absorbed energy values (KV) obtained are shown in Table 2 for each reference machine (identified by the acronyms SI, TO, TK), along with mean values (\overline{KV}), standard deviations (σ_{KV}), and coefficients of variation² (CV) for individual machines and for the whole group. Table 2 also reports the sample size, n_{SS} , calculated for Group A. The sample size is a statistical parameter that NIST uses in the certification of reference Charpy specimens to establish the acceptability of pilot and production lots: it corresponds to the number of specimens that a customer should test to obtain a statistically reliable comparison between their average absorbed energy and the NIST certified value. A pilot or production lot is considered acceptable if $n_{SS} \leq 5.0$ [6].

Table 2. Charpy test results for Group A.

Machine	KV (J)	Machine	KV (J)	Machine	KV (J)
SI	16.27	TO	17.26	TK	13.09
	17.48		16.05		14.70
	18.12		17.08		15.70
	15.78		17.08		14.50
	16.84		17.95		15.00
	17.49		16.14		14.20
	17.27		18.38		15.50
	17.69		18.12		14.50
	17.12		17.86		13.50
	18.34		18.20		15.30
Mean value (J)	17.24		17.41		14.60
Standard deviation (J)	0.82		0.83		0.83
Coefficient of variation	0.048		0.048		0.057
Overall mean:			$\overline{KV} = 16.42\text{ J}$		
Overall standard deviation:			$\sigma_{KV} = 1.53$		
Overall coefficient of variation:			$CV = 0.093$		
Sample size:			$n_{SS} = 5.247$		

² Coefficient of variation = ratio between standard deviation and mean value.

All broken specimens exited the machine backward at high speed, which basically removes the possibility of jamming (the likelihood of multiple collisions does not vanish, but becomes extremely low). Indeed, no outliers were identified by applying Grubbs' Test to group A.

2.2. Group B ($T_{temp} = 371\text{ }^{\circ}\text{C}$, $T_{test} = 21\text{ }^{\circ}\text{C}$)

Test results and statistical parameters for Group B are presented in Table 3. Specimens from this group underwent the same heat treatment as group A, but were tested at $21\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$.

Table 3. Charpy test results for Group B. The statistical parameters for the SI and TK machines, and for the whole group, were calculated after excluding the two outlier data points (highlighted in yellow).

Machine	KV (J)	Machine	KV (J)	Machine	KV (J)
SI	20.31	TO	22.14	TK	19.31
	21.47		19.88		18.80
	20.87		20.83		19.71
	20.30		21.35		20.31
	118.81		20.40		19.20
	21.96		21.53		19.20
	22.32		21.70		23.95
	23.77		20.75		19.51
	21.74		21.09		18.20
	22.89		19.01		19.81
Mean value (J)	21.74		20.87		19.34
Standard deviation (J)	1.16		0.92		0.61
Coefficient of variation	0.053		0.044		0.031
Overall mean:	$\overline{KV} = 20.66\text{ J}$				
Overall standard deviation:	$\sigma_{KV} = 1.33$				
Overall coefficient of variation:	$CV = 0.065$				
Sample size:	$n_{SS} = 6.185$				

Most specimens from group B dropped on the base of the machine, and only a few half-samples exited forward at moderate speed. These are favorable conditions for the occurrence of jamming and multiple collisions. Based on Grubbs' test, two values were identified as potential outliers (highlighted in yellow in Table 3): the 5th test on machine SI and the 7th test on machine TK. The former datum (118.81 J) is clearly the result of specimen jamming, while the latter might be the result of a minor jam, possibly coupled with post-fracture collisions.

2.3. Group C ($T_{temp} = 385\text{ }^{\circ}\text{C}$, $T_{test} = 21\text{ }^{\circ}\text{C}$)

Test results and statistical parameters for Group C are presented in Table 4.

The kinetics of Group C was similar to that of Group B, and the same number of high outliers (two) was flagged by Grubbs' test, one for the TO machine (9th test) and one for the TK machine (10th test). Moderate signs of jamming were observed on both specimens. As jammed

specimens have to be excluded from further analyses, the corresponding absorbed energy values were not used to calculate the statistics reported in Table 4.

Table 4. Charpy test results for Group C. The two outlier data points are highlighted in yellow.

Machine	KV (J)	Machine	KV (J)	Machine	KV (J)
SI	20.54	TO	20.19	TK	19.91
	20.31		20.79		18.00
	21.18		20.02		17.82
	19.81		20.45		18.42
	20.03		20.46		18.12
	19.88		20.62		17.72
	20.53		20.02		18.22
	20.53		20.19		18.02
	20.17		21.58		17.52
	20.10		20.11		20.86
	Mean value (J)		20.31		20.32
Standard deviation (J)	0.42	0.28	0.70		
Coefficient of variation	0.021	0.014	0.038		
Overall mean:	$\overline{KV} = 19.63 \text{ J}$				
Overall standard deviation:	$\sigma_{KV} = 1.11$				
Overall coefficient of variation:	$CV = 0.057$				
Sample size:	$n_{SS} = 2.232$				

2.4. Group D ($T_{temp} = 399 \text{ }^{\circ}\text{C}$, $T_{test} = 21 \text{ }^{\circ}\text{C}$)

Test results and statistical parameters for group D are presented in Table 5.

The specimens in group D, for the most part, were ejected forward (*i.e.*, in the direction of the pendulum swing) at low-to-moderate speed, and only in a few cases landed on the machine base. With this type of kinetics, the likelihood of jamming becomes low. No outliers were identified amongst the absorbed energy values. The average energy is slightly above the typical upper limit for low-energy specimens (20 J).

Table 5. Charpy test results for Group D.

Machine	KV (J)	Machine	KV (J)	Machine	KV (J)
SI	20.49	TO	21.56	TK	21.33
	21.51		21.73		20.12
	21.88		21.64		19.11
	20.85		22.34		20.02
	20.35		21.56		20.12
	21.43		21.47		19.92
	20.78		21.30		20.63
	21.80		22.60		20.93
	20.72		22.26		19.82
	21.59		22.34		20.22

Mean value (J)	21.14	21.84	20.22
Standard deviation (J)	0.52	0.46	0.64
Coefficient of variation	0.025	0.021	0.032
Overall mean:	$\overline{KV} = 21.07 \text{ J}$		
Overall standard deviation:	$\sigma_{KV} = 0.86$		
Overall coefficient of variation:	$CV = 0.041$		
Sample size:	$n_{SS} = 1.389$		

2.5. Group E ($T_{temp} = 413 \text{ }^{\circ}\text{C}$, $T_{test} = 21 \text{ }^{\circ}\text{C}$)

Test results and statistical parameters for Group E are presented in Table 6.

All specimens were ejected forward at moderate speed, and no outliers were identified among absorbed energy values. The average energy is significantly above the typical upper limit for low-energy specimens (20 J).

Table 6. Charpy test results for Group E.

Machine	KV (J)	Machine	KV (J)	Machine	KV (J)
SI	23.99	TO	25.14	TK	24.28
	23.19		26.54		22.16
	24.28		25.08		22.26
	24.44		24.82		22.26
	24.51		24.47		23.07
	23.77		24.56		22.77
	23.70		23.16		23.48
	24.28		23.85		22.97
	24.59		23.00		22.06
	23.55		24.47		22.47
Mean value (J)	24.03		24.68		22.81
Standard deviation (J)	0.46		0.93		0.69
Coefficient of variation	0.019		0.038		0.030
Overall mean:	$\overline{KV} = 23.77 \text{ J}$				
Overall standard deviation:	$\sigma_{KV} = 1.05$				
Overall coefficient of variation:	$CV = 0.044$				
Sample size:	$n_{SS} = 4.838$				

2.6. Group F ($T_{temp} = 343 \text{ }^{\circ}\text{C}$, $T_{test} = 21 \text{ }^{\circ}\text{C}$)

Test results and statistical parameters for group F are presented in Table 7.

Most specimens exited the machine backward at moderate-to-low speed and a few dropped on the base. Even though these are favorable conditions for jamming, no statistical outlier was identified among absorbed energy values.

Table 7. Charpy test results for group F.

Machine	KV (J)	Machine	KV (J)	Machine	KV (J)
SI	19.40	TO	19.10	TK	18.22
	18.82		19.27		17.22
	18.61		19.19		17.52
	17.82		18.75		17.82
	19.54		19.19		17.02
	19.32		18.75		16.92
	18.25		19.45		17.02
	18.39		18.75		17.82
	18.32		18.59		17.42
	18.46		18.67		17.52
Mean value (J)	18.69		19.01		17.44
Standard deviation (J)	0.51		0.29		0.41
Coefficient of variation	0.027		0.015		0.024
Overall mean:	$\bar{KV} = 18.37 \text{ J}$				
Overall standard deviation:	$\sigma_{KV} = 0.80$				
Overall coefficient of variation:	$CV = 0.043$				
Sample size:	$n_{SS} = 1.468$				

2.7. Group G ($T_{temp} = 316 \text{ }^{\circ}\text{C}$, $T_{test} = 21 \text{ }^{\circ}\text{C}$)

Test results and statistical parameters for group G, which corresponds to the lowest tempering temperature, are presented in Table 8.

All specimens exited the machine backward at high speed, and no outliers were identified among absorbed energy values. The average energy is right in the middle of the typical range for low-energy specimens (13 J to 20 J).

Table 8. Charpy test results for Group G.

Machine	KV (J)	Machine	KV (J)	Machine	KV (J)
SI	15.64	TO	18.52	TK	17.08
	18.20		18.43		16.68
	18.34		18.78		14.67
	17.13		16.71		14.87
	15.35		16.88		15.07
	15.28		16.03		15.47
	16.20		18.09		17.78
	18.56		18.70		16.98
	16.49		16.03		14.87
	15.85		18.52		16.58
Mean value (J)	16.70		17.85		15.94
Standard deviation (J)	1.25		1.03		1.00

Coefficient of variation	0.075	0.058	0.063
Overall mean:	$\overline{KV} = 16.79 \text{ J}$		
Overall standard deviation:	$\sigma_{KV} = 1.33$		
Overall coefficient of variation:	$CV = 0.079$		
Sample size:	$n_{SS} = 6.385$		

2.8. Group H ($T_{temp} = 371 \text{ }^\circ\text{C}$, $T_{test} = 21 \text{ }^\circ\text{C}$)

Group H had the same heat treatment and test temperature as group B, but specimens came from a different 4340 heat. Test results and statistical parameters for group H are presented in Table 9. Due to a malfunction of the acquisition system, only one absorbed value was recorded for the TK machine. This machine was therefore excluded from subsequent statistical calculations.

Table 9. Charpy test results for Group H.

Machine	KV (J)	Machine	KV (J)	Machine	KV (J)
SI	18.27	TO	19.19	TK	17.88
	18.78		19.10		
	19.64		19.02		
	18.64		18.24		
	18.49		19.62		
	17.42		20.14		
	19.21		18.93		
	18.57		19.36		
	18.85		18.93		
	18.63		19.45		
	Mean value (J)		16.70		17.85
	Standard deviation (J)		1.25		1.03
	Coefficient of variation		0.075		0.058
Overall mean:	$\overline{KV} = 16.79 \text{ J}$				
Overall standard deviation:	$\sigma_{KV} = 1.33$				
Overall coefficient of variation:	$CV = 0.079$				
Sample size:	$n_{SS} = 6.385$				

To confirm the equivalence between heats J3548 (groups A-E) and J2803 (groups F-H), a two-sample t-test assuming unequal variances was performed to test the hypothesis that the means of the 19 tests from group B conducted on the SI and TO machines (excluding the outlier datum) and of the 20 tests from group H are equal. The t-test results are summarized in Table 10. Since $t < t_{critical}$ and $p > \alpha = 0.05$, the hypothesis cannot be rejected, and the two heats can be considered equivalent. In subsequent analyses, test results from both groups/heats will be combined.

Table 10. Results of the t-test on the equivalence between SI and TO test results from groups B and H.

	Group B (heat J3548)	Group H (heat J2803)
Mean value (J)	20.11	20.03
Variance (J²)	3.27	1.26
Observations	19	20
Degrees of freedom	30	
<i>t</i>	0.163098	
<i>p</i> (two-tail)	0.871535	
<i>t</i>_{critical} (two-tail)	2.042272	
<i>α</i> (confidence level)	0.05	

3. Discussion

The statistical parameters of the 8 specimen groups (plus the combined B + H group) are summarized in Table 11, in ascending T_{temp} order.

Table 11. Statistical parameters for the specimen groups investigated.

Group	T_{temp} (°C)	T_{test} (°C)	\overline{KV}_{SI} (J)	\overline{KV}_{TO} (J)	\overline{KV}_{TK} (J)	\overline{KV} (J)	SD (J)	CV	n_{ss}	Statistical outliers	Post-fracture kinetics
G	316	21	16.70	17.85	15.94	16.79	1.33	0.079	6.385	0	B
F	343	21	18.69	19.01	17.44	18.37	0.80	0.043	1.468	0	B(Ba)
A	371	-40	17.24	17.41	14.60	16.42	1.53	0.093	5.247	0	B
B	371	21	21.74	20.87	19.34	20.66	1.33	0.065	6.185	2	B/Ba
H	371	21	18.65	19.23		18.87	0.63	0.033	1.551	0	B/Ba
C	385	21	20.31	20.32	18.19	19.63	1.11	0.057	2.232	2	F/Ba
D	399	21	21.14	21.84	20.22	21.07	0.86	0.041	1.389	0	F(Ba)
E	413	21	24.03	24.68	22.81	23.77	1.05	0.044	4.838	0	F
B+H	371	21	20.11	20.03	19.19	19.89	1.40	0.070	15.038	2	B/Ba

Legend

\overline{KV}_{SI} , \overline{KV}_{TO} , \overline{KV}_{TK} = mean values of absorbed energy for the SI, TO, and TK machine

\overline{KV} = mean value of absorbed energy for all machines

SD = standard deviation of absorbed energy for all machines

CV = coefficient of variation = SD/\overline{KV}

n_{ss} = sample size for all machines

Post-fracture kinetics B = all backward; B(Ba) = most backward, a few on base; B/Ba = some backward, some on base; F/Ba = some forward, some on base; F(Ba) = most forward, a few on base; F = all forward.

As the tempering temperature increases, the mean absorbed energy also increases. Concurrently, the post-fracture kinetics evolve from backward at high speed to forward at low/moderate speed, going through conditions where most or part of the broken specimens fall on the base of the machine, and are therefore at some risk of jamming. Mean absorbed energy values are shown in Fig. 2 as a function of tempering temperature for all machines combined, and in Fig. 3 for individual machines.

A strong positive correlation was found between T_{temp} and \overline{KV} , as evidenced by the high value of Pearson's linear correlation coefficient, $r = 0.939$. Conversely, negative correlations (parameter decreases with increasing T_{temp}), but quite weak, were found between T_{temp} and standard deviation ($r = -0.298$), coefficient of variation ($r = -0.637$), and sample size ($r = -0.132$).

The 21 °C test results that are most similar to the benchmark condition (group A, $T_{temp} = 371$ °C, $T_{test} = -40$ °C) correspond to group G ($T_{temp} = 316$ °C), both in terms of mean absorbed energy and post-fracture kinetics. This should therefore be the future tempering temperature for NIST low-energy specimens.

We also note from the results obtained (Table 11 and Fig. 3) that the TK machine produces systematically lower energy values than the SI and TO machines. This occurrence had already been observed in the past [8], and primarily depends on the machine/hammer design: the TK machine has a C-type hammer, while both SI and TO are U-type machines (Fig. 4). C-type machines are typically more rigid and less compliant than U-type machines, and therefore the

contribution of vibrational losses to the overall absorbed energy is less significant at the low-energy level. The higher rigidity of the TK machine with respect to SI and TO was clearly documented through specific compliance measurements [9].

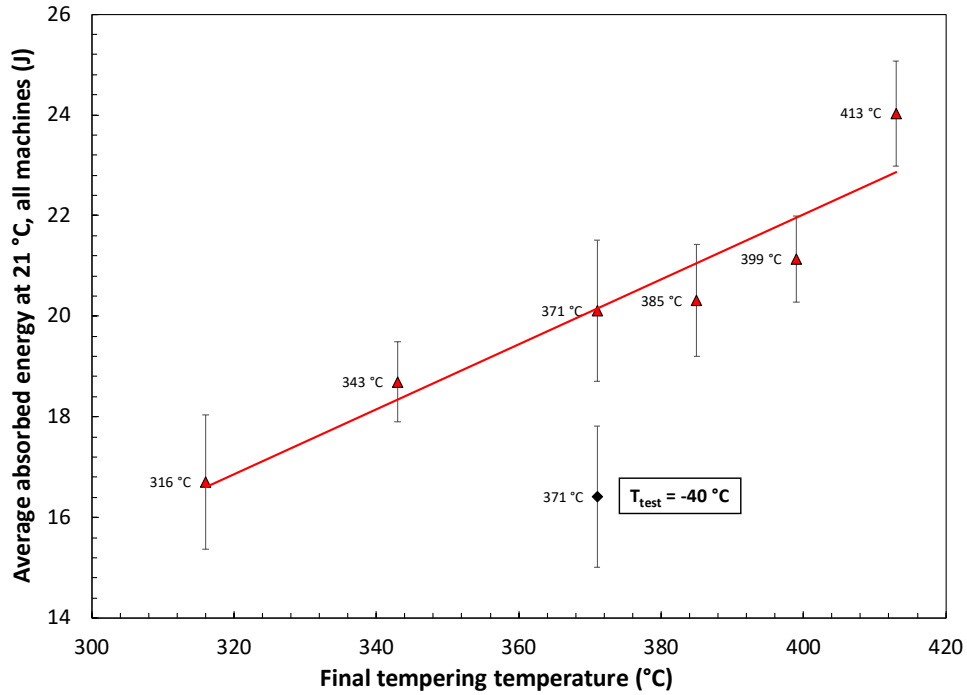


Fig. 2. Mean absorbed energy at 21 °C as a function of tempering temperature. Error bars correspond to standard deviations.

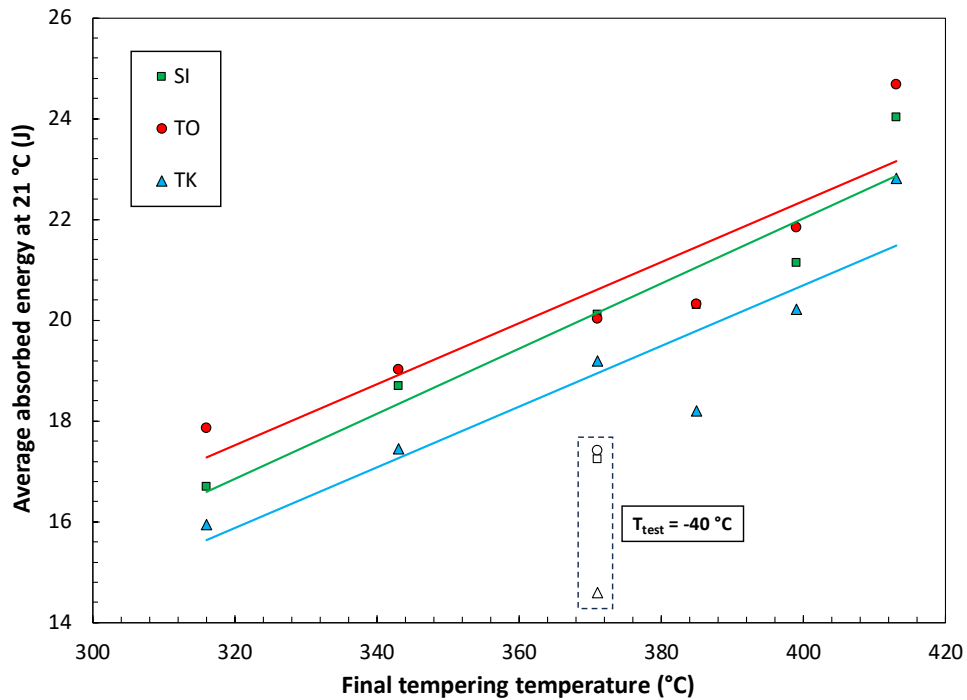


Fig. 3. Mean absorbed energy at 21 °C for the individual machines as a function of tempering temperature.

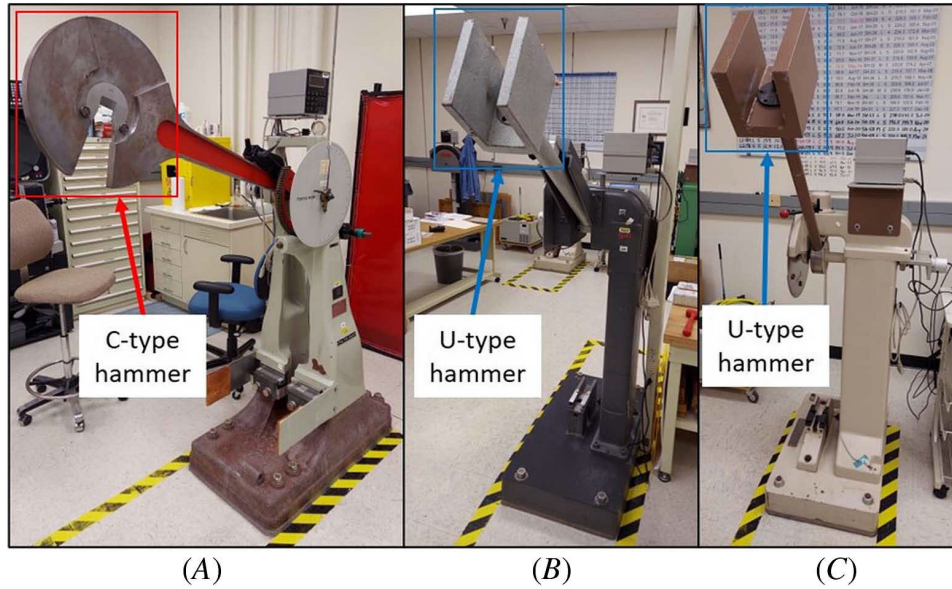


Fig. 4. The NIST Charpy reference machines: (A) C-type (TK) and (B), (C) U-type (TO and SI, respectively).

4. Conclusions

The investigations conducted on several groups of 4340 steel low-energy Charpy specimens, heat treated at different tempering temperatures, have unequivocally demonstrated that samples should be tempered at $T_{temp} = 316\text{ °C}/600\text{ °F}$ (lowest investigated temperature) to reproduce at $T_{test} = 21\text{ °C}$ the same testing conditions (absorbed energy and post-fracture kinetics) that occurred in the past when specimens were tested at -40 °C .

Specifically, the mean absorbed energy for $T_{temp} = 316\text{ °C}$ and $T_{test} = 21\text{ °C}$ was found to be only slightly higher than for $T_{temp} = 371\text{ °C}/700\text{ °F}$ (current tempering condition) and $T_{test} = -40\text{ °C}$, but the ± 1 standard deviation ranges largely overlapped. More importantly, the same post-fracture kinetics was recorded, with all specimens exiting the machine backward (opposite the hammer swing direction) at high speed, which appropriately minimizes the likelihood of specimen jamming or multiple collisions between broken specimens and test machine.

It is therefore recommended to temper at $316\text{ °C}/600\text{ °F}$ reference low-energy specimens for testing at 21 °C (SRM 2561, 2562, 2563, 2564, 2197).

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