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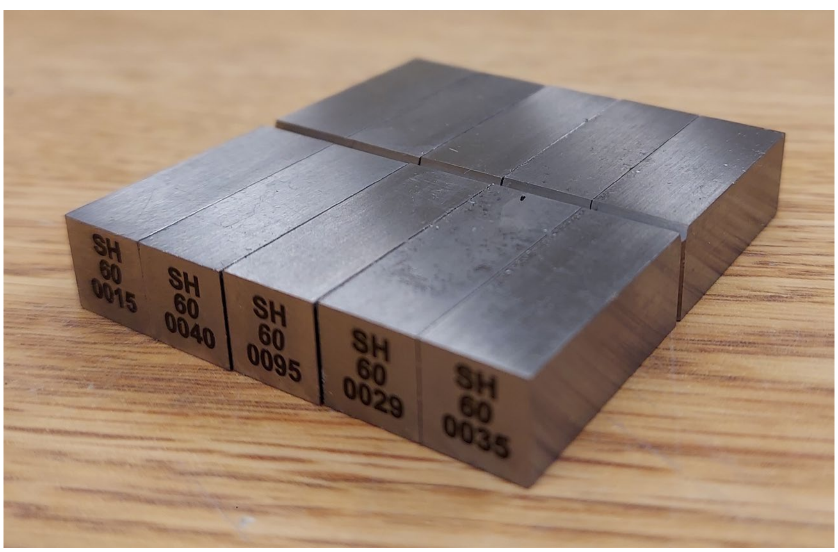

NIST NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
U.S. DEPARTMENT OF COMMERCE

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Influence of Final Tempering Temperature on Side-Grooved Super-High Energy Charpy Specimens of 9310 Steel (Second Phase)

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May 2023

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

NIST IR 8465
May 2023

NIST Technical Series Policies

Publication History

Approved by the NIST Editorial Review Board on 2023-05-01

How to Cite this NIST Technical Series Publication

Lucon E (2023) Influence of Final Tempering Temperature on Side-Grooved Super-High Energy Charpy Specimens of 9310 Steel (Second Phase). (National Institute of Standards and Technology, Boulder, CO), NIST Internal Report (IR) NIST IR 8465. https://doi.org/10.6028/NIST.IR.8465

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Public Comment Period

May 1, 2023 – May 1, 2024

Submit Comments

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Abstract

During Fiscal Year (FY) 2022, a study (presented in NIST IR8424) was conducted at NIST to identify the optimal combination of heat treatment (specifically, final tempering temperature) and side-groove depth for super-high energy reference Charpy specimens of 9310 steel. During FY23, a second phase of this investigation took place, wherein two additional values of the final tempering temperature were tested, while keeping the side-groove depth at the value previously established (0.9 mm/side, corresponding to 18 % of the original thickness). In consideration of the overall activities performed, final recommendations concerning the final tempering temperature and the side-groove depth are formulated for future NIST reference specimens of super-high energy level, manufactured from 9310 steel.

Keywords

9310 steel; ASTM E23; final tempering temperature; Rockwell C hardness; side-groove depth; super-high energy Charpy specimens.

Table of Contents

List of Tables

List of Figures

1. Introduction

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

Certified Charpy specimens are sold by NIST with three distinct energy levels: low-energy (13 J to 20 J at -40 °C), high-energy (90 J to 140 J at -40 °C), and super-high energy (175 J to 240 J at 21 °C). Samples from the first two energy levels are manufactured from ASTM/AISI 4340 steel, subject to different heat treatments, while the latter specimens are currently made of AISI 9310 steel. This steel has replaced the one used since the introduction of super-high energy specimens in the late 1990s, a 18Ni maraging steel denominated T-200. Investigations conducted prior to the reintroduction of super-high specimens into the NIST catalog[1] in 2018 showed that side-grooving of 9310 Charpy specimens was necessary to avoid a bimodal absorbed energy distribution, induced by the presence of shear lips on only one (asymmetrical fracture) or both (symmetrical fracture) broken specimen halves. **Fig. 1** illustrates the occurrence of asymmetrical and symmetrical fracture: the former is associated with significantly higher energy absorption than the latter [4]. As side-grooving inhibits the formation of shear lips, this bimodal behavior is avoided and the variability/scatter of test results is greatly reduced.

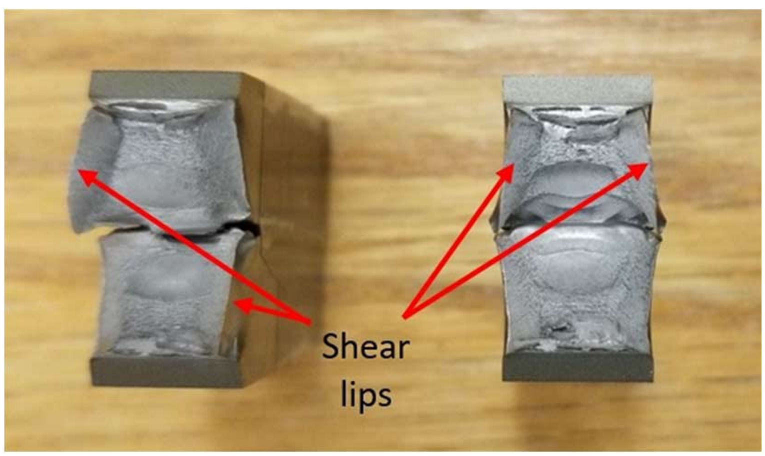

Fig. 1. Examples of asymmetrical (left) and symmetrical (right) fracture in Charpy specimens.

Two side-grove depths (0.9 mm/side and 1 mm/side) and three values of final tempering temperature, T_t (heat treatments A, B, and C) were investigated during FY22 [5]. While details of each specific heat treatment cycle are proprietary information of the company who heat treated and machined the specimens, T_t differed by 15 °F (8.3 °C) between A and B, and between B and C, with A corresponding to the lowest T_t and C to the highest T_t.

In the framework of the supplementary investigation reported here, the side-groove depth was maintained at 0.9 mm/side (1 mm/side was found to cause an excessive reduction of

[1] Super-high energy specimens went out of stock in 2008, and were unavailable to customers for approximately ten years.

absorbed energy), while two additional heat treatments were considered, D and E, with $T_{t(D)} = T_{t(C)} + 15$ °F and $T_{t(E)} = T_{t(D)} + 15$ °F.

The test matrix of the complete study (FY22-FY23) is summarized in **Table 1**.

Table 1. Overall test matrix for the activities performed in FY22 and FY23. *N* is the number of Charpy tests performed.

Heat Treatment	Side-groove depth (mm/side)	Side-groove depth %	FY	*N*
A	0.9	18	2022	150
	1	20		149
B	0.9	18		148
	1	20		145
C	0.9	18		148
	1	20		150
D	0.9	18	2023	75
E				74

The conclusions drawn from the FY22 activities can be summarized as follows:

- All six investigated combinations (three heat treatments and two side-groove depths) provided acceptable results in terms of sample size < 5.0.[2]
- No combination caused specimens to fully break on impact, due to the extreme ductility of the 9310 steel.
- The lowest sample size (heat treatment B, side-groove depth = 20 %) corresponded to an excessively low mean absorbed energy (166 J).
- The best compromise between low sample size (1.687) and acceptable mean absorbed energy (189 J) was found to correspond to heat treatment C (highest T_t) and 18 % side-grooving.

2. Charpy Tests Performed in FY23

As shown in **Table 1**, two additional conditions were investigated in the second part of this investigation, heat treatments D and E, the latter corresponding to the highest final tempering temperature (60 °F = 33.3 °C higher than heat treatment A). For both conditions, side-grooving corresponded to 0.9 mm/side or 18 % of the original thickness.

For both conditions, 75 Charpy specimens were tested (25 on each NIST reference machine). All tests were performed at room temperature (21 °C ± 1 °C). The absorbed energy, *KV*, results are presented in **Table 2** (heat treatment D) and **Table 3** (heat treatment E). Rockwell C hardness measurements, *HRC*, were also conducted on 10 specimens per condition (**Table 4**).

[2] The sample size for a NIST Charpy specimen lot is given by:

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

with 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. The current acceptance criterion for a NIST certified lot is $n_{SS} < 5.0$.

Table 2. Charpy absorbed energies obtained for heat treatment D. SI, TO, and TK identify the three NIST ASTM E23 reference machines.

NIST machine	*KV* (J)	NIST machine	*KV* (J)	NIST machine	*KV* (J)
SI	201.47	TO	204.33	TK	207.68
	206.27		202.74		204.68
	201.65		205.74		207.12
	198.23		202.92		209.55
	204.89		210.91		207.59
	204.98		218.52		210.85
	207.56		201.68		206.46
	204.52		200.17		213.07
	198.69		214.47		212.42
	193.96		212.48		206.09
	194.52		206.00		208.80
	197.67		213.52		206.65
	200.73		207.50		209.45
	208.76		205.30		207.49
	205.72		206.18		214.27
	205.44		210.65		211.31
	202.02		210.47		212.33
	205.90		211.08		208.33
	205.26		194.10		207.87
	202.30		202.56		208.52
	202.58		203.45		210.29
	197.77		198.66		209.92
	197.77		207.94		206.74
	194.33		200.26		206.18
	205.44		196.51		209.73

Table 3. Charpy absorbed energies obtained for heat treatment E.

NIST machine	*KV* (J)	NIST machine	*KV* (J)	NIST machine	*KV* (J)
SI	215.05	TO	210.04	TK	225.65
	212.94		222.03		239.54
	208.62		220.15		220.32
	208.71		217.67		213.36
	209.26		212.65		220.69
	214.22		204.42		230.72
	206.04		217.92		226.81
	206.77		219.13		224.57
	206.77		218.52		216.12
	201.32		218.61		212.90
	208.99		215.51		217.68
	208.71		218.44		215.48
	217.43		218.87		221.41
	206.77		205.74		217.50
	206.22		222.80		217.50
	209.26		203.62		222.59
	215.32		218.10		230.64
	212.76		213.52		221.78
	207.79		211.26		229.57
	205.85		218.95		222.86
	206.96		210.39		229.40
	208.43		218.44		233.63
	204.56		220.75		223.31
	203.17		217.15		215.76
			216.37		219.23

Table 4. Rockwell C hardness measurement results.

Heat treatment	HRC	Heat treatment	HRC
D	22.25 22.40 22.20 22.45 22.35 22.10 22.60 22.35 22.40 22.55	E	20.75 21.60 21.50 21.80 21.50 21.40 21.35 21.40 21.15 21.30

Overall statistics for the eight conditions investigated in FY22 and FY23 are summarized in **Table 5**. For hardness, only mean values ($\overline{HRC}$) are provided.

Table 5. Overall absorbed energy statistics for the 8 conditions investigated.

Heat treatment	Side-grooving (%)	$\overline{KV}$ (J)	σ_{KV} (J)	CV[3] (%)	$\overline{HRC}$	Sample size, n_{SS}
A	18	178.96	4.61	2.6	25.30	2.097
	20	166.15	3.87	2.3		1.348
B	18	187.07	4.71	2.5	23.79	1.879
	20	166.41	2.45	1.5		0.670
C	18	189.41	4.49	2.4	23.08	1.687
	20	186.09	5.31	2.9		2.996
D	18	205.60	5.25	2.6	22.37	1.684
E	18	215.84	7.86	3.6	21.38	2.320

3. Discussion

The results obtained in FY22 and FY23 allow illustrating the trends of mean absorbed energy $\overline{KV}$ (**Fig. 2**), mean hardness $\overline{HRC}$ (**Fig. 3**), and sample size n_{SS} (**Fig. 4**) as a function of heat treatment or T_t.

Clear trends were observed for both absorbed energy and hardness, respectively increasing and decreasing with increasing final tempering temperature for both side-groove depths. Consequently, and as expected, absorbed energies and hardness are also strongly correlated (**Fig. 5**).

Sample sizes (representing the variability of the Charpy test results) exhibit a minimum at intermediate values of T_t for both side-grooving depths, but remain excellent (less than 2.5) for all conditions examined. This confirms that the current production process of super-high energy Charpy specimens of 9310 steel is robust and delivers satisfactory results.

Mean absorbed energies are above the minimum ASTM E23 recommended level (176 J) for shallower side-grooves (0.9 mm/side), while they fall below the threshold for the two lower values of T_t (heat treatments A and B) in case of deeper side-grooves (1 mm/side). Predictably, deeper side-grooves cause a reduction in absorbed energy.

[3] The Coefficient of Variation, *CV*, is given by the ratio between standard deviation and mean value, expressed in percentage.

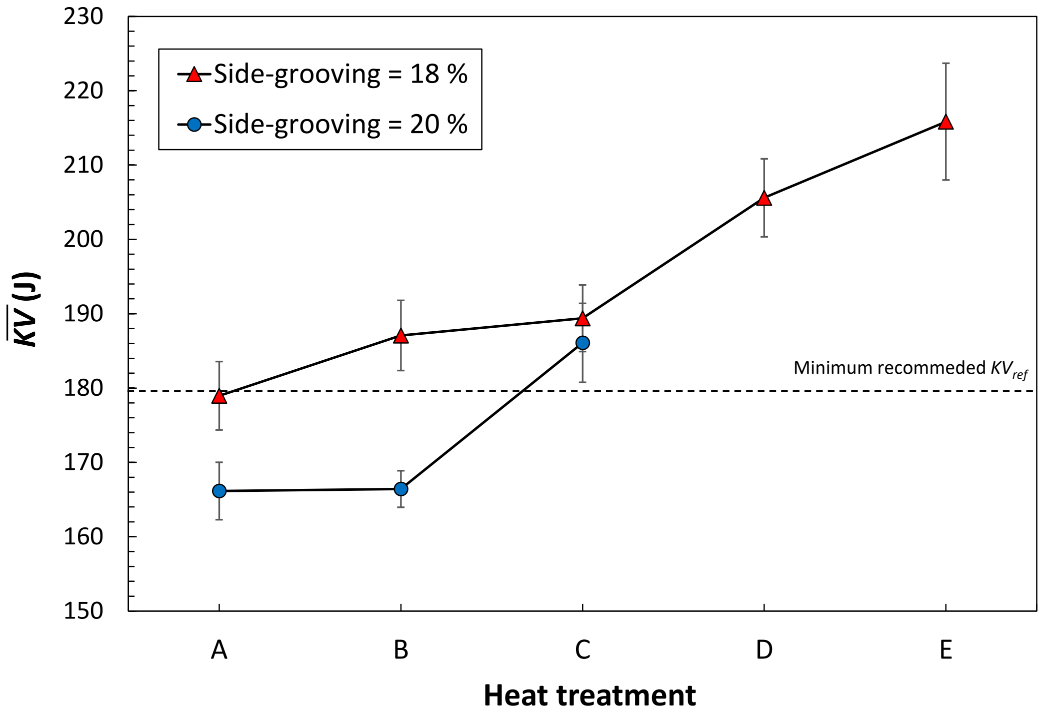

Fig. 2. Mean absorbed energies obtained for the different heat treatments and side-groove depths. Error bars correspond to one standard deviation.

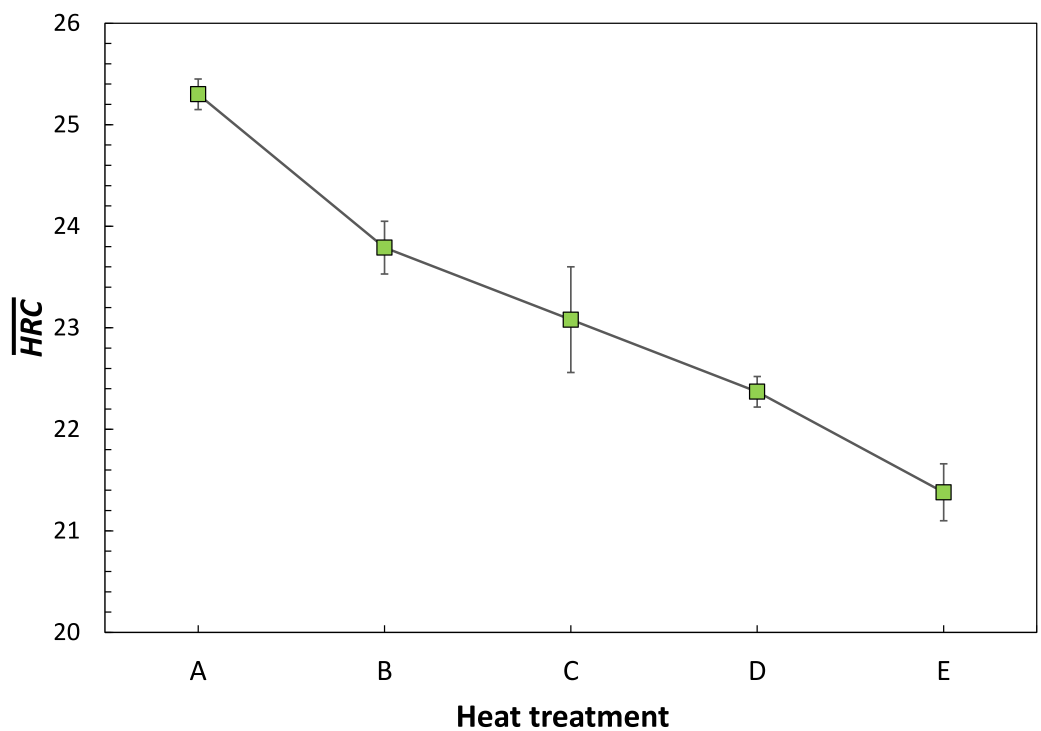

Fig. 3. Mean Rockwell C hardness values obtained for the different heat treatments and side-groove depths. Error bars correspond to one standard deviation.

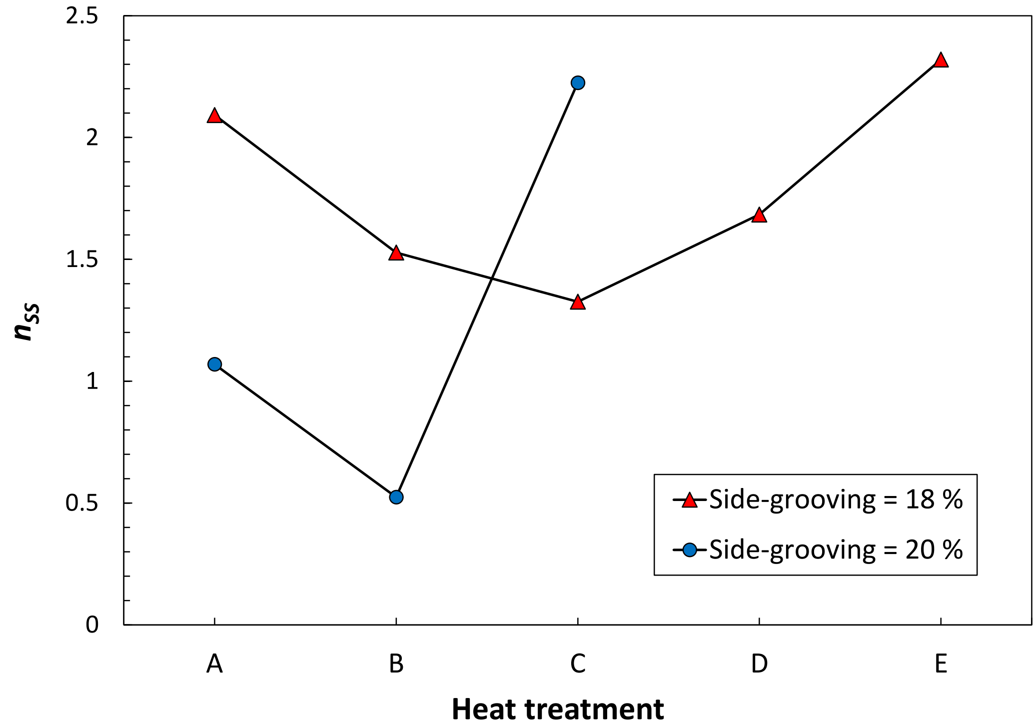

Fig. 4. Sample sizes calculated for the different heat treatments and side-groove depths.

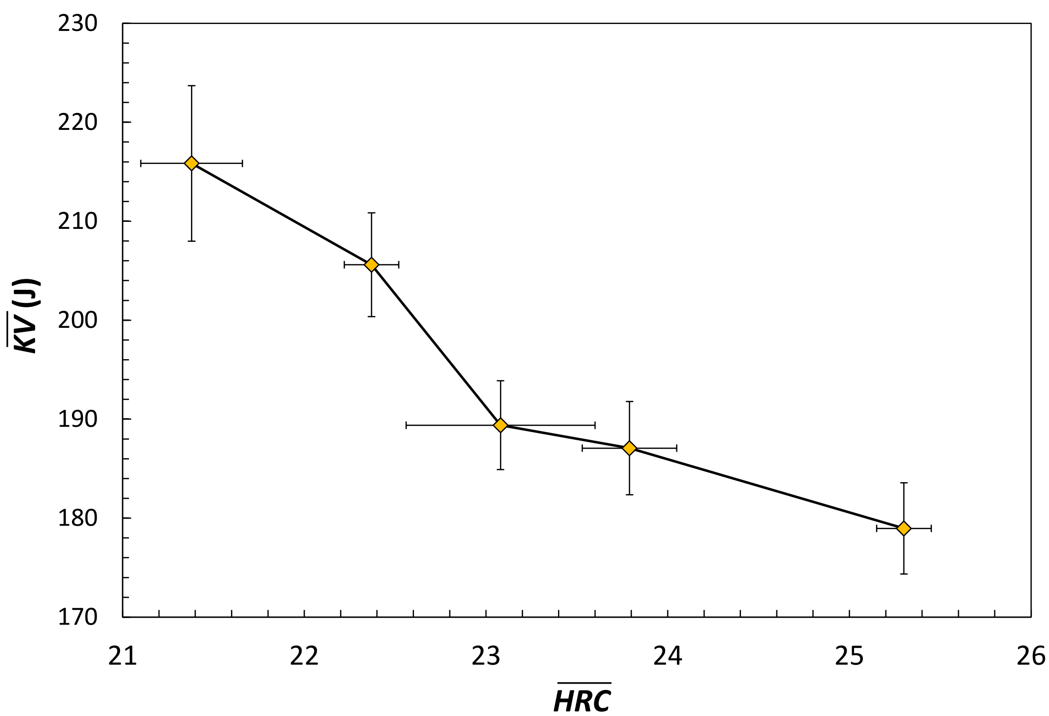

Fig. 5. Relationship between hardness and absorbed energy for the investigated heat treatments. Error bars correspond to one standard deviation.

4. Conclusions

In FY22 and FY23, a two-part investigation was conducted at NIST aimed at establishing the optimal combination of heat treatment (specifically, final tempering temperature T_t) and side-groove depth for super-high energy Charpy specimens made of 9310 steel for the Charpy Machine Verification Program.

Eight combinations of heat treatment (five different values of T_t) and side-groove depth (0.9 mm/side and 1 mm/side) were investigated, by testing at room temperature a total of 1039 specimens on the three ASTM E23 Charpy reference machines in Boulder, Colorado. All combinations provided largely acceptable results according to NIST specifications (sample size < 5.0). All tested specimens remained unbroken, due to the high ductility of the steel.

Strong and monotonic correlations were observed between final tempering temperature and mean absorbed energy and hardness, as well as between absorbed energy and hardness. The best combination between sample size (1.684) and mean absorbed energy (205.6 J) corresponds to heat treatment D 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.

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