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INVESTIGATION OF RELATIONSHIP BETWEEN MICROHARDNESS AND CHARPY IMPACT ENERGY FOR TEMPER BEAD WELDING QUALIFICATION: PART 1

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ABSTRACT

Temper bead (TB) welding is often used as an alternative to post weld heat treatment (PWHT) for repair of pressure vessels and piping in the nuclear power industry. Historically, qualification of TB welding procedures has employed the Charpy V-notch test to ensure acceptable heat-affected-zone (HAZ) impact properties. The 2004 Edition of ASME Section IX provided a new provision in QW-290 that allows temper bead qualification using a peak hardness criterion. The peak hardness provision is appropriate for industries such as oil and gas, where peak allowable hardness is specified to ensure adequate resistance to sulfide stress cracking in sour service environments. However, a peak hardness criterion is not appropriate where impact properties are specified for resistance to brittle fracture during low temperature conditions that can occur during certain postulated accident scenarios at a nuclear power plant.

Work at the Electric Power Research Institute (EPRI) and The Ohio State University (OSU) show that a hardness drop protocol can be used to demonstrate acceptable impact properties in the HAZ of a temper bead weld. This paper presents a quantitative correlation between hardness measurements and HAZ microstructures with presumed optimum impact properties using a hardness drop approach. The overarching goal is to develop a hardness test protocol for temper bead weld procedure qualification for applications where impact properties are specified.

NOMENCLATURE

CVN	Charpy V-notch
GND	Geometrically Necessary Dislocations
HAZ	Heat Affected Zone
HV	Vickers Hardness
ISE	Indentation Size Effect
TB	Temper Bead

INTRODUCTION

Temper bead (TB) welding is a common alternative to post weld heat treatment (PWHT) where PWHT is determined to be impractical or not possible. ASME Section XI, IWA-4600 and Code Cases, such as Case N-638-9 [1], specify Charpy V-notch (CVN) impact testing to qualify temper bead welding procedures. A temper bead welding procedure is successfully qualified when the CVN impact energy and lateral expansion properties in the test plate HAZ are equal to or better than the unaffected base material properties. It is important to understand that the CVN test does not measure material fracture toughness but rather demonstrates adequate fracture toughness by an empirical relationship with CVN impact properties [2].

The 2004 Edition of ASME Section IX provided a new provision in QW-290 that allows temper bead qualification using a peak hardness criterion [3]. With some limited exceptions, like NACE Standard MR0103 [4] for sour service applications, codes and standards used in the nuclear power industry do not specify peak hardness acceptance criteria for weldments following PWHT or temper bead welding. Of even greater significance is the fact that a single peak hardness criterion is not appropriate for demonstrating acceptable HAZ impact properties. Reference [5] discusses this issue in detail. A summary of the issues with applying a single peak hardness criterion are noted below:

- Peak hardness alone, without knowing the microstructure or thermal history, is not adequate to verify appropriate HAZ tempering.
- Use of peak hardness criterion can potentially lead to acceptance of temper bead procedures that generate a HAZ microstructure with poor impact properties.
- Rejection of a temper bead procedure qualification by a single peak hardness reading (as often required in EN/ISO codes) is neither reasonable nor appropriate.

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PROPOSED TWO STEP HARDNESS PROTOCOL FOR TEMPER BEAD PROCEDURE QUALIFICATION

In 2014 EPRI proposed an alternative temper bead procedure qualification protocol using a two-step Vickers microhardness approach [7, 8]. A fundamental premise of the protocol is that an effective temper bead welding procedure will first develop a high percentage of untempered Martensite in the HAZ. This Martensite is then properly tempered by the heat from adjacent weld beads and weld layers. As discussed in detail in References [2] and [6], tempered Martensite is the desired microstructure to achieve optimum CVN impact properties.

Step 1 of the proposed EPRI two-step protocol is to perform a single bead-on-plate test on a material of the same P-No and Group No to be welded using the temper bead welding parameters. The purpose of this single bead-on-plate step is to characterize the hardness response of the base material with the temper bead parameters. Step 2 of the protocol is to weld a three layer pad on the same material with the temper bead welding parameters. The aim of the three layer pad is to demonstrate the tempering effectiveness of the temper bead procedure. An appropriate drop in hardness between the un-tempered HAZ with the single bead-on-plate (Step 1 in Figure 1) and the tempered HAZ following the three layer temper bead weld pad (Step 2 in Figure 1) demonstrates the tempering effectiveness of the temper bead welding procedure.

Figure 1 below illustrates Step 1 with high hardness and low impact energy of un-tempered Martensite and Step 2 with low hardness and high impact energy. The tempered and untempered specimens of Bainite and Martensite HAZ microstructures depicted in Figure 1 were made with vintage A-508 forged base material using a Gleeble® thermo-mechanical weld simulator.

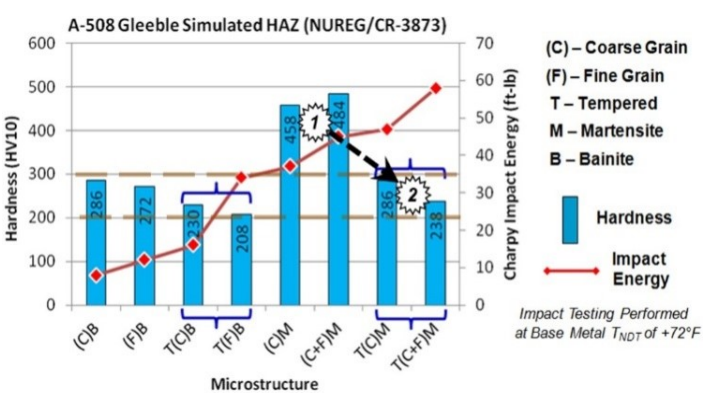

Figure 1: *Steps 1 and 2 show hardness drop between untempered and tempered Martensite for Gleeble simulated A-508 HAZ samples [7, 8]. Note that tempered Martensite with good impact energy and both untempered and tempered Bainite with poor impact energy are in the same hardness range.*

Using the impact energy and hardness values for the coarse and fine grain tempered and untempered Martensite in Figure 1, a pass-fail hardness drop relationship to demonstrate acceptable impact energy is illustrated in Figure 2. The vertical line at 150 HV hardness drop depicts a possible acceptance criterion.

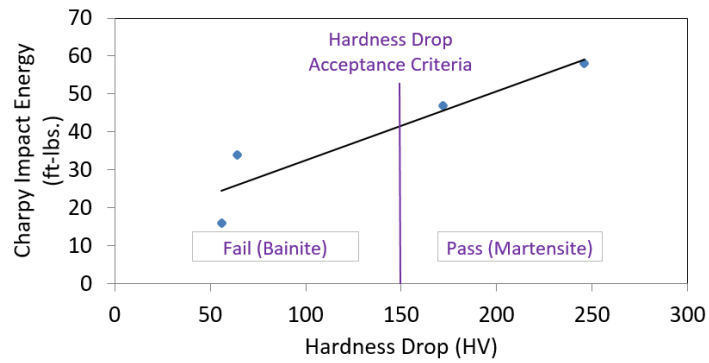

Figure 2: *Example of tempered impact energy versus hardness drop with pass-fail line (data adapted from Figure 1).*

Relevant issues presented in this paper to support the proposed EPRI two step hardness drop protocol for temper bead welding procedure qualification are:

- Optimization of Vickers microhardness load and spacing for SA-508 HAZ microstructures.
- Influence of mixed phase HAZ microstructures of Bainite and Martensite on untempered and tempered hardness.
- Quantitative hardness drop values that demonstrate effective tempering of HAZ microstructures with expected high CVN impact energy.

The material selected for evaluation was SA-508 Grade 2 Class 1, a common reactor pressure vessel (RPV) quenched and tempered forging material used in the nuclear industry. The composition of the SA-508 material is listed in Table 1.

Table 1: *Composition (wt %) of pressure vessel forge material used to investigate hardness drop approach in this paper.*

Material	SA-508 Gr. 2 Cl. 1
C	0.2
Mn	0.61
P	0.01
S	0.009
Si	0.24
Cu	--
Ni	0.68
Cr	0.32
Mo	0.59
V	0.01
Ti	--
Al	--

HARDNESS TESTING BACKGROUND

Vickers microhardness testing of metallic materials is mainly governed by ASTM Standards E92 [9] and E384 [10]. The main operational takeaways from these two standards are:

- The spacing between indentations as well as from the edge of the sample must be 2.5 times the mean length of the indentation, dV, shown in Figure 3.
- Any indentation made with a load of 1000 grams force or higher can be considered a bulk hardness measurement.

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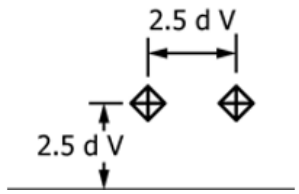

Figure 3: *Minimum recommended spacing for Vickers microhardness indents [8].*

If the spacing between indentations is too small it can lead to a greater dislocation density due to the adjacent indent and will lead to elevated hardness values. If the indentation is made too close to the edge of the sample this will lead to insufficient material to constrain the indent which will lead to depressed hardness values.

A load of 1000 grams force or larger is considered a bulk hardness measurement because of the indentation size effect (ISE). The ISE occurs because whenever an indentation is made material must be deformed and moved causing geometrically necessary dislocations (GND) to be produced under the indenter tip [11]. This gives a higher dislocation density under the indenter tip than that of the bulk material, and this effect is exaggerated at smaller indentation depths with smaller loads. The ISE is shown in Figure 4.

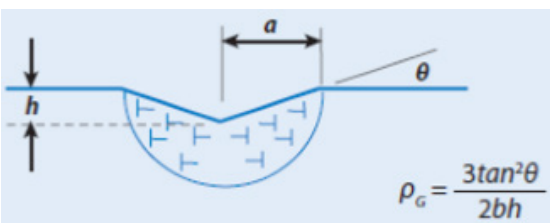

Figure 4: *Nix and Gao model for conical indenters with geometrically necessary dislocation (GND) density (ρ_G) equation where θ is dependent on the geometry of the indenter, b is Burger's vector, and h is the indentation depth [9].*

HARDNESS TESTING PARAMETER OPTIMIZATION

A series of statistical analyses were performed to investigate optimum Vickers microhardness testing parameters for SA-508 base material and for SA-508 HAZ microstructures after TB Welding. The first step was a statistical analysis on SA-508 Gr. 2 Class 1 base material. A total of 100 indents were taken at loads varying from 10 to 1000 grams with indentation spacing well above the 2.5 d-spacing required by the ASTM standards [9, 10]. As the data in Table 2 show, the hardness values were 264 HV with a 10 gram load as compared to the bulk hardness measurement of 208 HV with a 1000 gram load.

The data in Table 2 also show that a 200 gram load is optimum with the least statistical scatter since the 95% confidence is 1.05 (lowest of all other loads). It is important to understand that the optimum load for HAZ microstructures with higher hardness will be higher to achieve low data scatter with a similarly low 95% confidence value.

Table 2: *Statistical hardness analysis confirm indentation size effect (ISE) for SA-508 Gr.2 Cl.1 base material.*

Load (g)	Hardness (HV)	Std. Dev.	95% Confidence	Mean Length (microns)
10	264	14	2.7	8.4
25	236	9.1	1.8	14.0
50	222	6.4	1.25	20.4
100	213	5.9	1.16	29.5
200	210	5.3	1.05	42.0
300	211	7.4	1.45	51.2
500	206	6.8	1.33	67.2
1000	208	8.4	1.64	94.4

Load and spacing were the two parameters to be optimized for measuring the SA-508 HAZ hardness since it is known that they can affect the hardness values and variability of the measurements [9, 10]. The different microstructures in the HAZ of a TB weld were simulated using a Gleeble 3800 thermo-mechanical simulator. Simulated cooling rates were set at 10°, 20°, and 40° C/s. Tempering temperature was set at 635° C with hold times of 1, 10, 100 seconds, and no tempering for each of the simulated cooling rates. These simulated materials were then hardness tested with loads varying from 100, 200, 500, and 1000 grams with hardness indent spacing of 1.5, 2.5, 5.0, and 7.5 dV.

A total of over 40,000 indents were made and measured to create a statistical hardness study of mainly 10 by 10 indentation matrices using a Leco LM100-AT automated Vickers microhardness indenter. Intermittent results were used to formulate each of the new design of experiments for hardness testing with the end goal of identifying optimized hardness testing parameters.

Based on the statistical study, a 500 gram load is recommended because it gave the smallest indentation that was also a bulk hardness measurement for all SA-508 HAZ microstructures investigated. The 500 gram load hardness measurements were within one standard deviation of a bulk hardness measurement at 1000 gram load for each of the studied microstructures.

Two sample t–statistic tests were performed on the hardness of the tempered Martensite sample shown in Table 3. The t-statistic test is a type of inferential statistic used to determine if there is a significant difference between the means of two groups and gives a confidence level to determine whether or not two samples come from the same population. The t – statistic relationship is:

$$t - statistic = \frac{\bar{x}_1 + \bar{x}_2}{s/\sqrt{n}}, \quad (1)$$

where $\bar{x}_1$ and $\bar{x}_2$ are the average hardness values for the different loads, s is the average standard deviation of the two data sets, and n is the sample size.

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The t – statistic test was done for 200 gram with 1000 gram load measurements and 500 gram with 1000 gram load measurements at each indent dV spacing for the tempered martensitic microstructure (40° C/s cooling rate with a 1 second temper time at 635° C).

Table 3: *SA-508 Gr.2 Cl.1, HAZ thermal cycle, 40º C/s cooling rate with a 1 second temper at 635º C from the statistical hardness study.*

Load (grams)	Spacing (µm)	D-Spacing (300 HV)	Average Hardness (HV)	Standard Deviation
100	38	1.5 * D	359	12.9
100	63	2.5 * D	340	10.0
100	125	5.0 * D	342	12.6
100	188	7.5 * D	335	12.2
200	53	1.5 * D	363	26.2
200	88	2.5 * D	333	8.7
200	175	5.0 * D	342	12.6
200	263	7.5 * D	335	13.1
500	84	1.5 * D	325	7.0
500	140	2.5 * D	324	8.1
500	280	5.0 * D	327	10.8
500	420	7.5 * D	325	10.1
1000	119	1.5 * D	325	8.0
1000	198	2.5 * D	320	8.3
1000	395	5.0 * D	327	10.0
1000	593	7.5 * D	328	8.2

The t – statistic results were found to be 30 for the 200 gram and 1000 gram load measurements and 0.56 for the 500 gram and 1000 gram load measurements using equation (1). This means that the data sets for the 200 gram and 1000 gram loads come from a different population at a 95 percent confidence level because their t-statistic of 30 is greater than the critical t-value of 1.97 for a 95% confidence level at a population of 400. Conversely, the 500 gram and 1000 gram loads come from the same population at a 95 percent confidence level since their t – statistic of 0.56 is less than the critical t-value of 1.97. A similar trend was seen for the rest of the simulated microstructures in the statistical study.

The recommended spacing between hardness indents can be further optimized after looking into the untempered hard region size, however, based on the t – statistic test results the recommended spacing between indentations would be 250 µm to stay within ASTM E92 and E384 Standards. The 250 micron spacing is considered a conservative spacing parameter because hard regions in similar TB weldments at 800 microns or even larger did not have a detrimental effect on impact properties [5].

HARDNESS DROP VALUES AND PHASE FRACTION ANALYSIS

Hardness drop values were determined by measuring the average hardness of the untempered and tempered microstructures from the statistical hardness study. Tables 4 and 5 list the cooling rates, % Martensite (M), % Bainite (B), untempered hardness, tempered hardness and hardness drop with 500 gram load and 1000 gram load, respectively. Phase fraction analysis was performed using optical manual quantitative metallography of Nital etched micrographs. The phase fraction analysis was supported by a MATLAB macro that superimposed the point counting grid onto the micrographs to speed up the process. It is important to note there is a distinct difference between the lathy feature of untempered Martensite and the primarily untempered upper (fine grain) Bainite structure. Figure 5 shows a side by side comparison of untempered upper Bainite cooled at 5° C/s and untempered Martensite cooled at 55° C/s.

The data in Tables 4 and 5 show that the hardness drop is in the range of 112 to 132 HV for microstructures with greater than 90% Martensite (cooling rates of 30° C/s, 40° C/s, and 55° C/s). The data also show there is a strong linear correlation between the untempered percentages of Martensite to the hardness drop that occurs with tempering. Figure 6 plots the percentage of Martensite in the untempered microstructure to the hardness drop after tempering for both the 500 and 1000 gram load data. Note that the R^2 regression correlation coefficient is greater than 0.9 for both the 500 and 1000 gram load data indicating a strong linear relationship. Based on tempered Martensite having superior impact properties compared to other microstructures [2, 6] and the hardness drop proving to be an accurate predictor of starting percentage of Martensite (Table 4, Table 5, and Figure 6), the hardness drop is a strong indicator of good CVN impact energy values.

Table 4: *Hardness drop with 500 gram load for varying phase fractions of Martensite (M) and Bainite (B). Phase fractions were determined by quantitative metallography. Cooling rates are 800º C to 500º C from austenization at 969º C for 5 minutes.*

Cooling Rate	M%	B%	Std. Dev	Untempered Hardness (HV 0.5)	Hardness after 1s tempering at 635ºC (HV 0.5)	Hardness Drop (HV 0.5)
5 C/s	19	81	9	305 ± 2	276 ± 1	29 ± 2
8 C/s	28	72	17	353 ± 2	302 ± 1	51 ± 2
10 C/s	60	40	17	386 ± 3	307 ± 2	80 ± 3
15 C/s	84	16	5	432 ± 2	329 ± 1	103 ± 2
20 C/s	77	23	12	419 ± 2	325 ± 1	94 ± 2
30 C/s	93	7	4	455 ± 1	330 ± 1	126 ± 1
40 C/s	91	9	4	441 ± 2	329 ± 1	112 ± 2
55 C/s	99	1	1	462 ± 1	342 ± 1	120 ± 1
SA-508 BM	NA	NA	NA	207 ± 8	NA	NA

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Table 5: *Hardness drop with 1000 gram load for varying phase fractions of Martensite (M) and Bainite (B). Phase fractions were determined by quantitative metallography. Cooling rates are 800º C to 500º C from austenization at 969º C for 5 minutes.*

Cooling Rate	M%	B%	Std. Dev.	Untempered Hardness (HV 1.0)	Hardness after 1s tempering at 635ºC (HV 1.0)	Hardness Drop (HV 1.0)
5 C/s	19	81	9	303 ± 2	269 ± 1	34 ± 2
8 C/s	28	72	17	354 ± 2	295 ± 1	59 ± 2
10 C/s	60	40	17	395 ± 2	291 ± 1	104 ± 3
15 C/s	84	16	5	432 ± 2	328 ± 1	104 ± 2
20 C/s	77	23	12	422 ± 2	324 ± 1	98 ± 3
30 C/s	93	7	4	455 ± 1	323 ± 1	132 ± 1
40 C/s	91	9	4	453 ± 1	327 ± 1	127 ± 1
55 C/s	99	1	1	458 ± 1	336 ± 1	123 ± 1
SA-508 BM	NA	NA	NA	205 ± 1	NA	NA

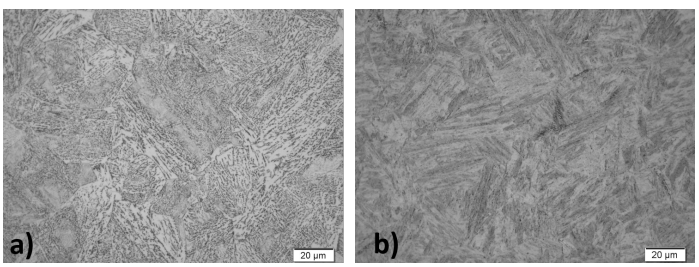

Figure 5: *Micrographs of a) primarily untempered upper Bainite (19% M and 81% B) cooled at 5º C/s and b) untempered Martensite (99% M and 1% B) cooled at 55º C/s. Both with austenization of 969º C for 5 minutes, Nital etched at 1000x.*

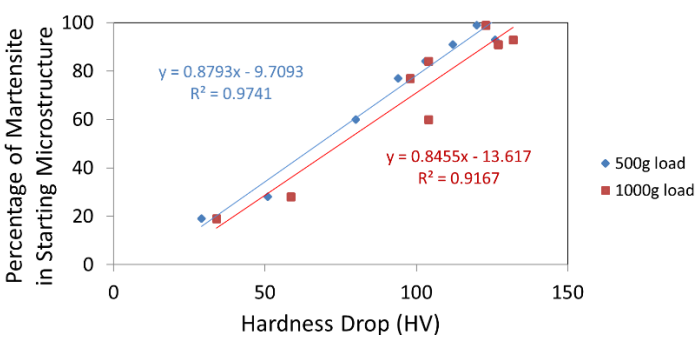

Figure 6: *Correlation between percentage of Martensite in the untempered microstructure to hardness drop after tempering (data are from Tables 4 and 5).*

PASS – FAIL APPROACH THAT DEMONSTRATES MICROSTRUCTURE WITH SATISFACTORY LEVEL OF TEMPERERED MARTENSITE

The data in Tables 4 and 5 show that if a point hardness above 440 HV is reached in the untempered condition, greater than 90% Martensite is in the HAZ microstructure. The data also show that 90% Martensite is achieved with cooling rates of 30° C/s or greater. A drop in point hardness of 110 HV or greater from the 440 HV peak threshold was adequate to demonstrate proper tempering. If a peak point hardness of 440 HV is not reached in the untempered microstructure, a hardness drop of 125 HV or greater was adequate to demonstrate proper tempering. Using this relationship, a two part pass-fail hardness drop criterion can be applied to evaluate the effectiveness of tempering. A flow chart depicting this approach is shown in Figure 7. It is important to note that a basic assumption of this approach is that a high phase fraction of tempered Martensite is the optimum microstructure for high CVN impact energy.

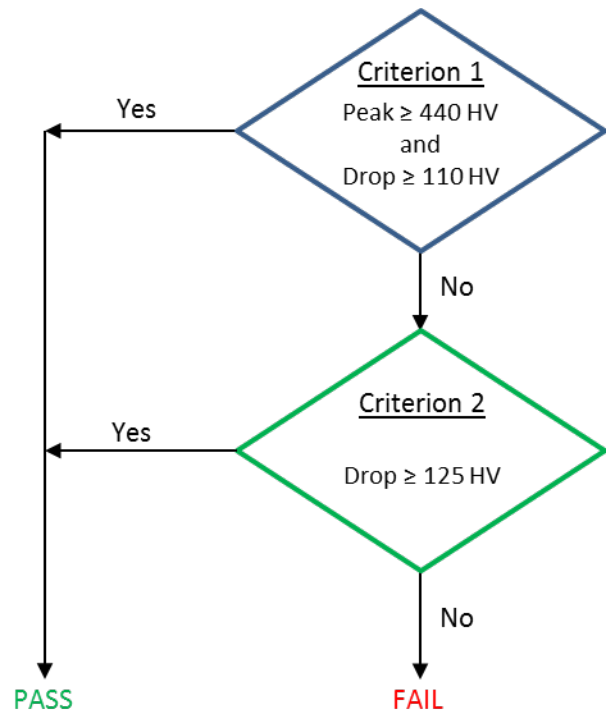

Figure 7: *Flowchart using peak hardness and hardness drop measurements as indicators of appropriate level of tempered Martensite. Criterion 1 and Criterion 2 come from data primarily obtained in Tables 4 and 5 respectively.*

The pass-fail hardness criterion in Figure 7 was cross-checked by investigating the untempered and tempered response of a Jominy bar of the same SA-508 heat of material. A Jominy end quench was performed in accordance with ASTM A255 [13]. Instead of performing Rockwell hardness testing, Vickers microhardness with a 500 gram load and 250 µm spacing along 5 traverses with 80 indents total were made down the length of the as-quenched Jominy bar. Similar hardness traverses on the same Jominy bar after a heat treatment at 635° C for 1 hour were then performed. Short tempering time at a peak temperature typical for TB welding are achievable with a Gleeble but are not feasible for the Jominy bar. The 1 hour tempering is however considered reasonable for cross-checking the pass-fail criterion. The Vickers microhardness measurements from the as-quenched and tempered Jominy bar were then processed through the pass-fail criterion. The results are plotted in Figure 8.

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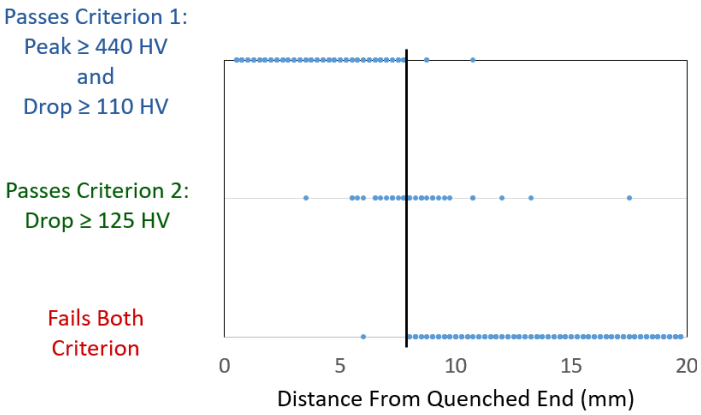

Figure 8: *SA-508 Gr. 2 Class 1 Jominy bar hardness traverses evaluated according to pass-fail criterion in Figure 7.*

Figure 8 shows that failures in accordance with pass-fail Criterion 1 start around 8 mm from the quenched end of the Jominy bar (vertical black line in Figure 8). This corresponds to a cooling rate of about 40° C/s, which is the critical cooling rate for formation of Martensite in SA-508 material based on experimental results [6, 8] and computational CCT diagrams calculated using JMatPro®. In the region between 8 and 10 mm some failure points occur, but also a significant number of points pass Criterion 2. The reason for this is that the 8 to 10 mm region corresponds to cooling rates between 40° C/s to 30° C/s, which have 90% or greater Martensite mixed with Bainite based on the point count analysis (Tables 4 and 5). Points that pass Criteria 2 drop off rapidly past 10 mm and only 5 out of 200 points pass either criterion past the 10 mm distance. Cooling rates beyond 10 mm on the Jominy bar are 20° C/s or lower where the mixed microstructure has a decreasing percentage of Martensite and increasing percentage of Bainite (Tables 4 and 5). This analysis shows that the proposed peak hardness with hardness drop criterion outlined in Figure 7 demonstrates a predominately tempered martensitic structure, indicating an optimal microstructure with good CVN impact energy.

SUMMARY AND CONCLUSIONS

Vickers microhardness testing with a 500 gram load and spacing of 250 µm were found to be optimum parameters for hardness evaluation of temper bead HAZ of SA-508 Gr. 2 Class 1 alloy steel forging material. This result is based on statistical analysis of a large matrix of hardness measurements at different loads and indent spacing on simulated HAZ microstructures over a range of heat treatments. It is also important to note that hardness measurements using a 1000 gram load were not significantly different in hardness or variability from measurements using a 500 gram load.

Evaluation of Gleeble simulated mixed Bainite-Martensite microstructure hardness data show that the magnitude of hardness drop that occurs with tempering, as well as the peak hardness prior to tempering, has a strong correlation to the percentage of Martensite in the tempered microstructure. The data also show that 90% or greater Martensite occurred with cooling rates of 30° C/s or higher. Cooling rates greater than 30° C/s or greater are typical for a good temper bead procedure.

The data in Tables 4 and 5 show that for the heat of SA-508 material tested, the hardness drop is in the range of 112 to 132 HV for microstructures having a martensitic phase fraction greater than 90%. Evaluation of this hardness drop criterion was shown to accurately predict the amount of tempered Martensite in the simulated HAZ microstructure. The hardness drop criterion was further evaluated by comparing Vickers microhardness along a Jominy bar made the of the same SA-508 material in the as quenched condition and following a heat treatment of 635º C for 1 hour. The cooling rates and hardness drop values for the Jominy results correspond closely with the Gleeble simulated HAZ data.

Finally, the data suggest a strong linear relationship between the untempered percentages of Martensite to the hardness drop that occurs with tempering. This linear relationship can be used to correlate the hardness drop to CVN impact energy.

The EPRI proposed two part TB weld qualification criterion was demonstrated with the Gleeble simulated samples. The data show that an acceptable level of tempered Martensite is achieved for untempered peak hardness points above 440 HV with a minimum hardness drop of 110 HV (Criterion 1) or for untempered peak hardness points below 440 HV with a minimum hardness drop of 125 HV (Criterion 2). Since the hardness drop of a single point in the HAZ of a qualification weld is not possible in actual application a conservative criterion invoking either Criterion 1 or Criterion 2, or a combination, would be applied for the EPRI proposed two step TB qualification protocol.

FUTURE WORK

Part 2 of this work will evaluate the CVN impact energy for simulated SA-508 HAZ microstructures in the untempered and tempered condition. The goal is to develop a correlation for the percentage of Martensite in the untempered and tempered HAZ microstructures with CVN impact energy.

Fracture toughness will also be evaluated for each of the simulated microstructures using instrumented indentation with a Frontics AIS 3000. These fracture toughness results will then be compared with the CVN impact energy results and hardness drop analysis.

Finally, a SA-508 Gr. 3 Class 2 forging, as well as two different heats of ASTM A-387 F22 commonly used in the oil and gas industry, will be evaluated against the hardness drop criterion.

ACKNOWLEDGEMENTS

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