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Hydrogen Embrittlement Susceptibility of Bainite for High Strength Steel Fasteners

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Executive Summary

With the objective to save weight and to render designs more fuel efficient, the approach of increasing fastener strength while reducing weight is being explored, notably in the automotive industry. The most concerning challenge in using higher strength steel fasteners is their increased susceptibility to hydrogen embrittlement (HE). As strength increases, so does susceptibility to HE, thus increasing the risk of in-service failure. The engineering objective of hydrogen embrittlement research in the field of mechanical fasteners is to find and develop steel alloys and microstructure for high strength and ultrahigh strength fasteners that are less susceptible to HE failure than conventional alloys.

In the current work, a comparative study of HE susceptibility was performed on two different microstructures, martensite and lower bainite. Comparisons were made at near-equal strength using screw samples made of AISI 8640 grade steel. The samples were cold formed and heat treated, quench and tempered to obtain martensite, or austempered to obtain lower bainite, by industrial partners who manufacture and supply fasteners to the auto industry. Incremental step load (ISL) testing was the method used for measuring HE threshold stress under varying cathodic hydrogen charging conditions. The findings indicate the more ductile lower bainite samples exhibit marginally lower HE susceptibility when tested under moderate hydrogen charging conditions (e.g., -1.0 V). At the most severe hydrogen charging potential of -1.2 V both microstructures are equally embrittled. These findings are consistent with other proprietary studies and are likely explained by the transport and trapping kinetics of hydrogen in bainite as compared to martensite [1].

Another finding is that cold forming the threads (i.e., thread rolling) after heat treatment had a significant effect of increasing HE threshold for both microstructures. The data show the beneficial effect of thread rolling after heat treatment outweighs the marginal benefit obtained by only changing the microstructure to bainite. This important finding is likely related to a significant increase in dislocation density resulting from cold work in the hardened threads. Dislocations are reversible traps that can reduce H transport kinetics by increasing H trapping. The outcome is that less hydrogen becomes locally available to participate in H damage mechanisms.

In summary, the results of this study show that changing to lower bainitic structures for manufacturing ultrahigh strength fasteners only has a marginal effect of reducing HE susceptibility. In comparison, thread rolling after heat treatment has a significantly greater effect of reducing HE susceptibility for both martensite and lower bainite. These findings are presented strictly in the context of hydrogen embrittlement susceptibility. This study does not claim to discount other potential improvements in mechanical properties that may be obtained from lower bainitic structures, such as higher impact energy and increased yield strength, especially at tensile strengths above 1,200 MPa.

For the purpose of this summary report, the data are presented in the following pages without full commentary. Footnotes are given to elaborate on the most important experimental details.

Reference – limited to the scope of the Executive Summary

1. ***Impact de la structure métallurgique sur le chargement et la désorption naturelle de L'hydrogène dans un acier (37Cr4) traite à 1250MPa***, A. Fleurentin (Cetim), J. Creus et X. Feaugas (LaSIE), Cetim, Paris, 2013.

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Respectfully submitted,

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USCAR – IFI: Result Summary
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Sample Description

<table>
<tr><th>Supplier</th><th colspan="3">ITW</th><th colspan="4">MNP</th></tr>
<tr><td>Sample type</td><td colspan="7">Hex head flange bolt</td></tr>
<tr><td>Sample size</td><td colspan="3">M8-1.25X80mm</td><td colspan="4">M8-1.25X30mm</td></tr>
<tr><td>Microstructure</td><td colspan="2">Tempered martensite</td><td>Lower bainite</td><td colspan="2">Lower bainite</td><td colspan="2">Tempered martensite</td></tr>
<tr><td>Stated hardness</td><td colspan="2">46 HRC</td><td>48 HRC</td><td>39 HRC</td><td>44 HRC</td><td>39 HRC</td><td>44 HRC</td></tr>
<tr><td>Condition</td><td colspan="3">Rolled before HT / unplated</td><td colspan="4">Rolled after HT / unplated</td></tr>
<tr><td>Sample ID</td><td>B3</td><td colspan="2">B4</td><td>B6</td><td>B8</td><td>B7</td><td>B9</td></tr>
</table>

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Sample condition- Chemical

Sample Code	Micro	%C	%Cr	%Mn	%Mo	%Ni
B3	Martensite	0.39	0.45	0.80	0.15	0.45
B4	Bainite	0.40	0.44	0.80	0.15	0.44
B6	Bainite	0.44	0.49	0.94	0.18	0.48
B7	Martensite	0.44	0.50	0.94	0.18	0.50
B8	Bainite	0.44	0.50	0.95	0.18	0.49
B9	Martensite	0.44	0.50	0.94	0.18	0.49
UNS G86400 Requirement		0.38-0.43	0.40-0.60	0.75-1.00	0.15-0.25	0.40-0.70

Evaluation by ICP and Combustion

1 sample per batch

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Sample condition- Chemical

Sample Code	Micro	%P	%S	%Si	Hydrogen (ppm)
B3	Martensite	0.007	0.012	0.20	0.6
B4	Bainite	0.006	0.014	0.20	3.4
B6	Bainite	0.010	0.002	0.22	2.0
B7	Martensite	0.011	0.002	0.22	0.8
B8	Bainite	0.012	0.002	0.22	2.5
B9	Martensite	0.011	0.002	0.22	1.9
UNS G86400 Requirement		0.035 max	0.040 max	0.15-0.35	-

Evaluation by ICP and Combustion

1 sample per batch

Note 1: Chemical analysis showed the bainitic materials contained higher total hydrogen concentrations in the as-received samples.

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Sample condition- Hardness

Sample Code	Supplier	Microstructure	Mid-radius Hardness (HRC)*	Sub-Surface hardness (HRC)* at 0.002” below root
B3	ITW	Martensite	45	47
B4	ITW	Bainite	47	47
B6	MNP	Bainite (RAHT)	39	49
B7	MNP	Martensite (RAHT)	39	48
B8	MNP	Bainite (RAHT)	44	52
B9	MNP	Martensite (RAHT)	44	50

5 samples per batch evaluated

Note 2: RAHT: rolled after heat treatment
Note 3: Sub-surface hardness results showed a significant increase in hardness near the surface generated by the thread rolling after heat treatment (samples B6 to B9).
Note 4: Rolling after heat treatment caused greater increase in surface hardness at 0.002” below the thread root in the bainitic samples.

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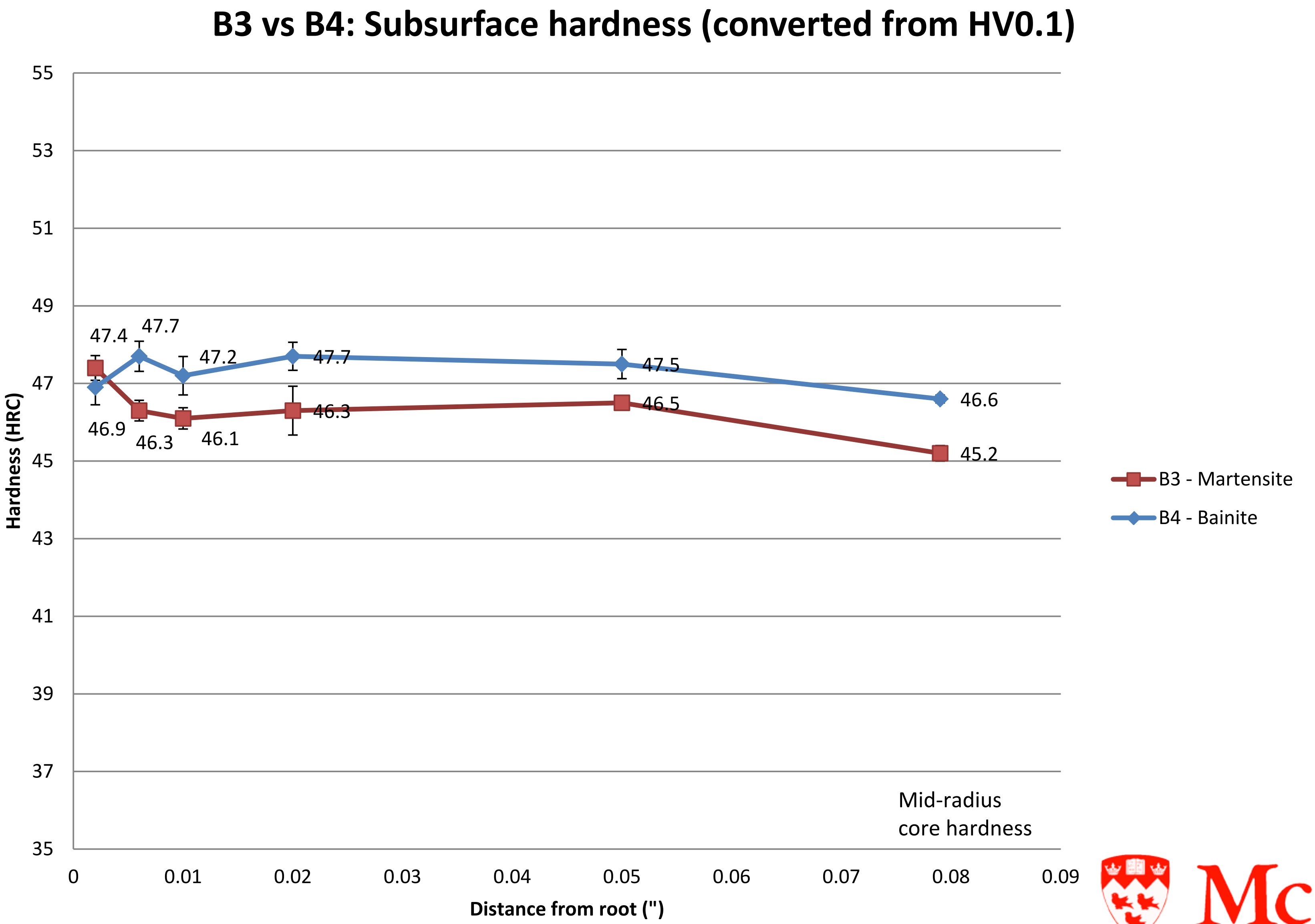
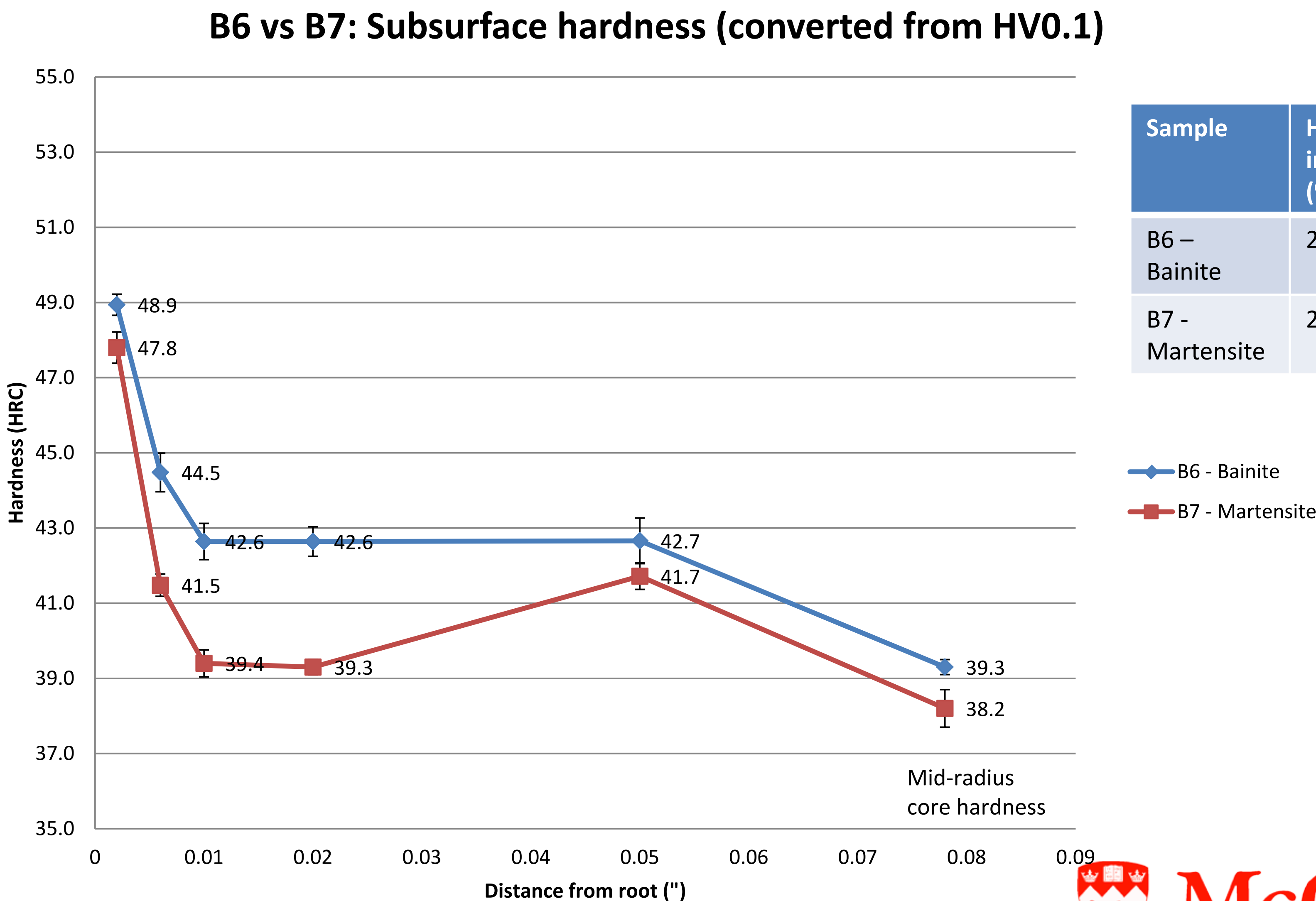
Sample condition- Hardness



Sample condition- Hardness



Sample	HV increase (%)
B6 – Bainite	25.6
B7 - Martensite	23.1

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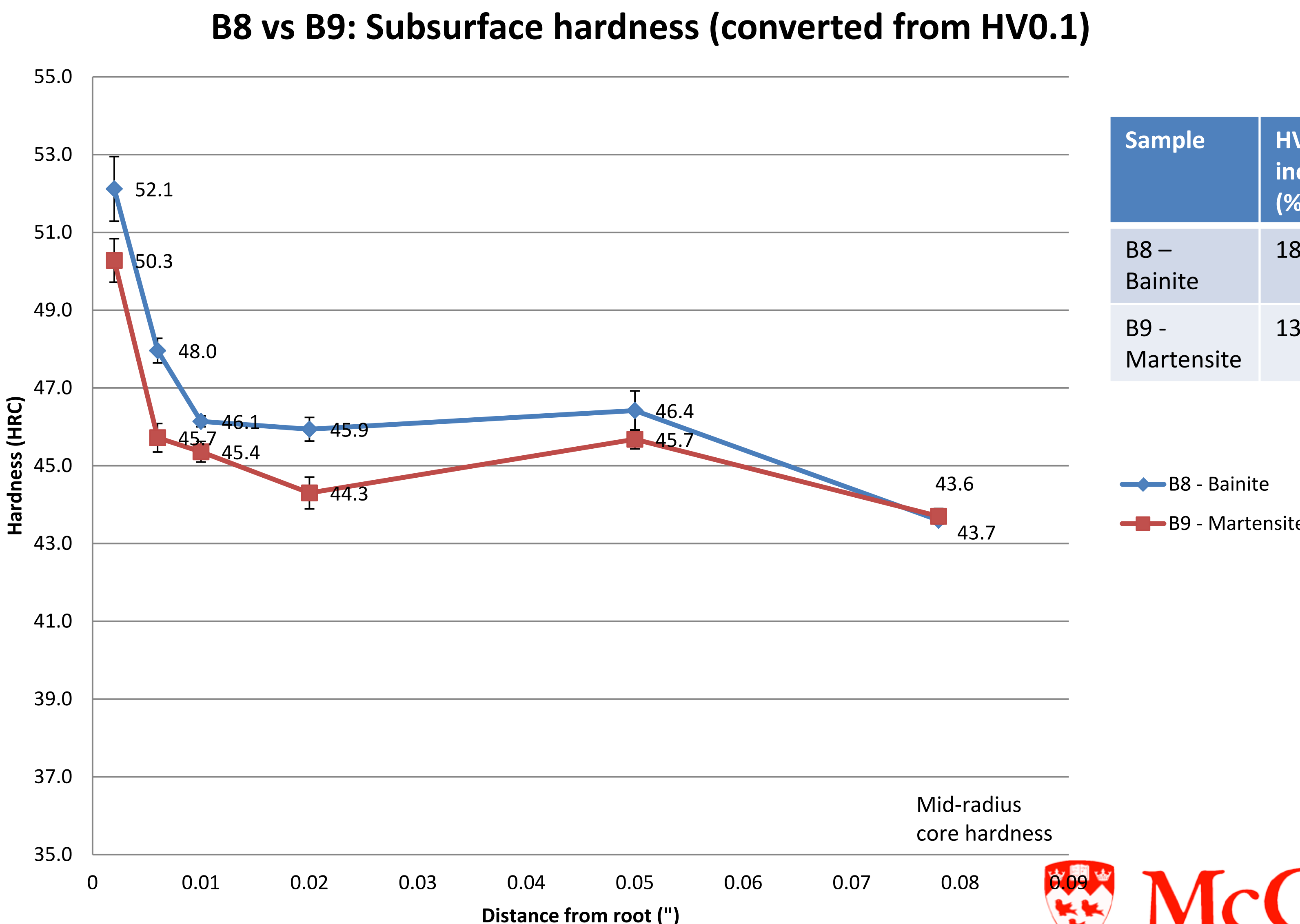
Sample condition- Hardness



Sample	HV increase (%)
B8 – Bainite	18.1
B9 - Martensite	13.6

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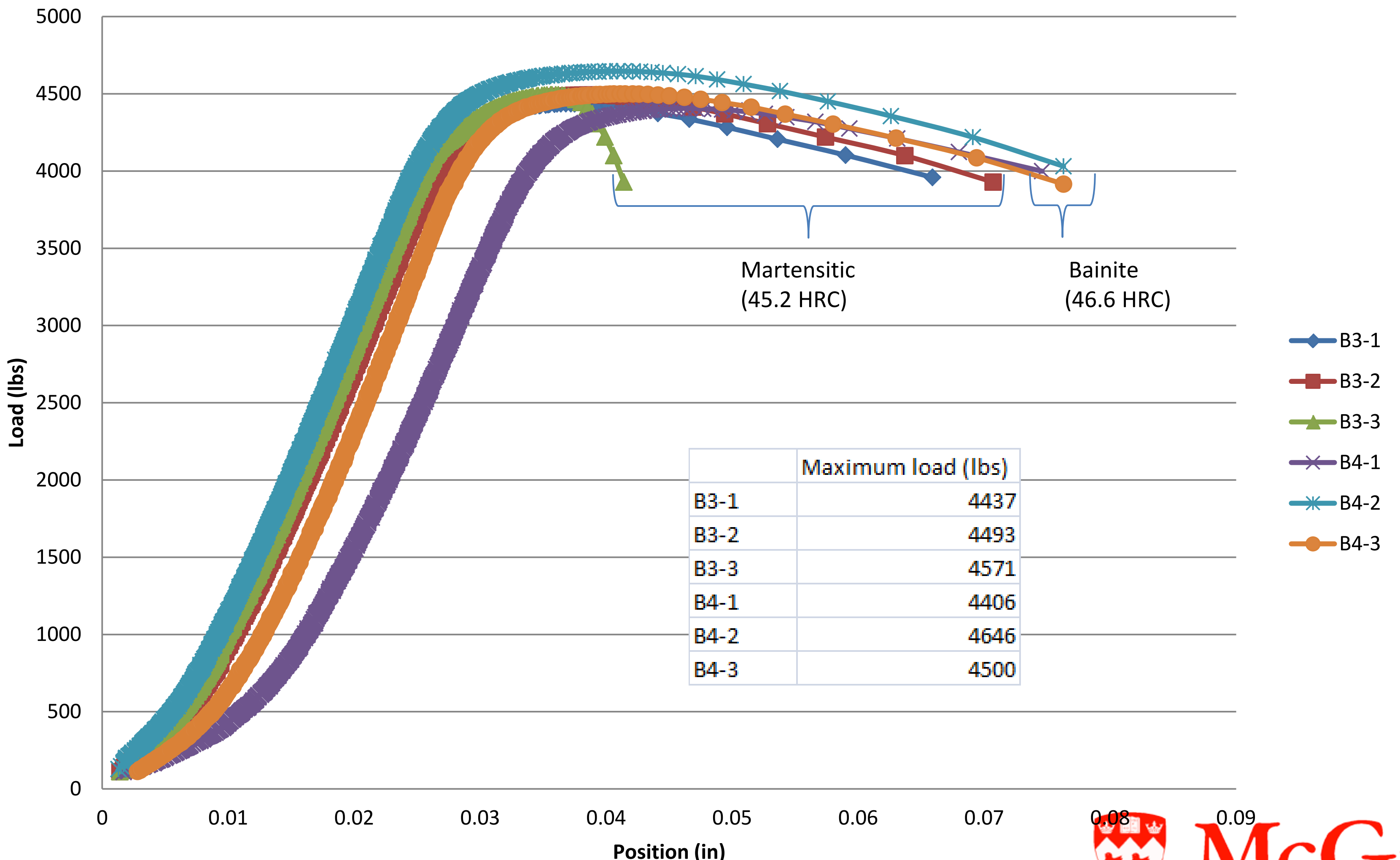
Sample condition- Tensile

Samples machined from screws based on
ASTM E8-13a Figure 8 modified Specimen 4

* 3 samples per batch evaluated per E8

Sample condition- Tensile

B3 (Martensite) vs B4 (Bainite) Tensile Evaluation



	Maximum load (lbs)
B3-1	4437
B3-2	4493
B3-3	4571
B4-1	4406
B4-2	4646
B4-3	4500

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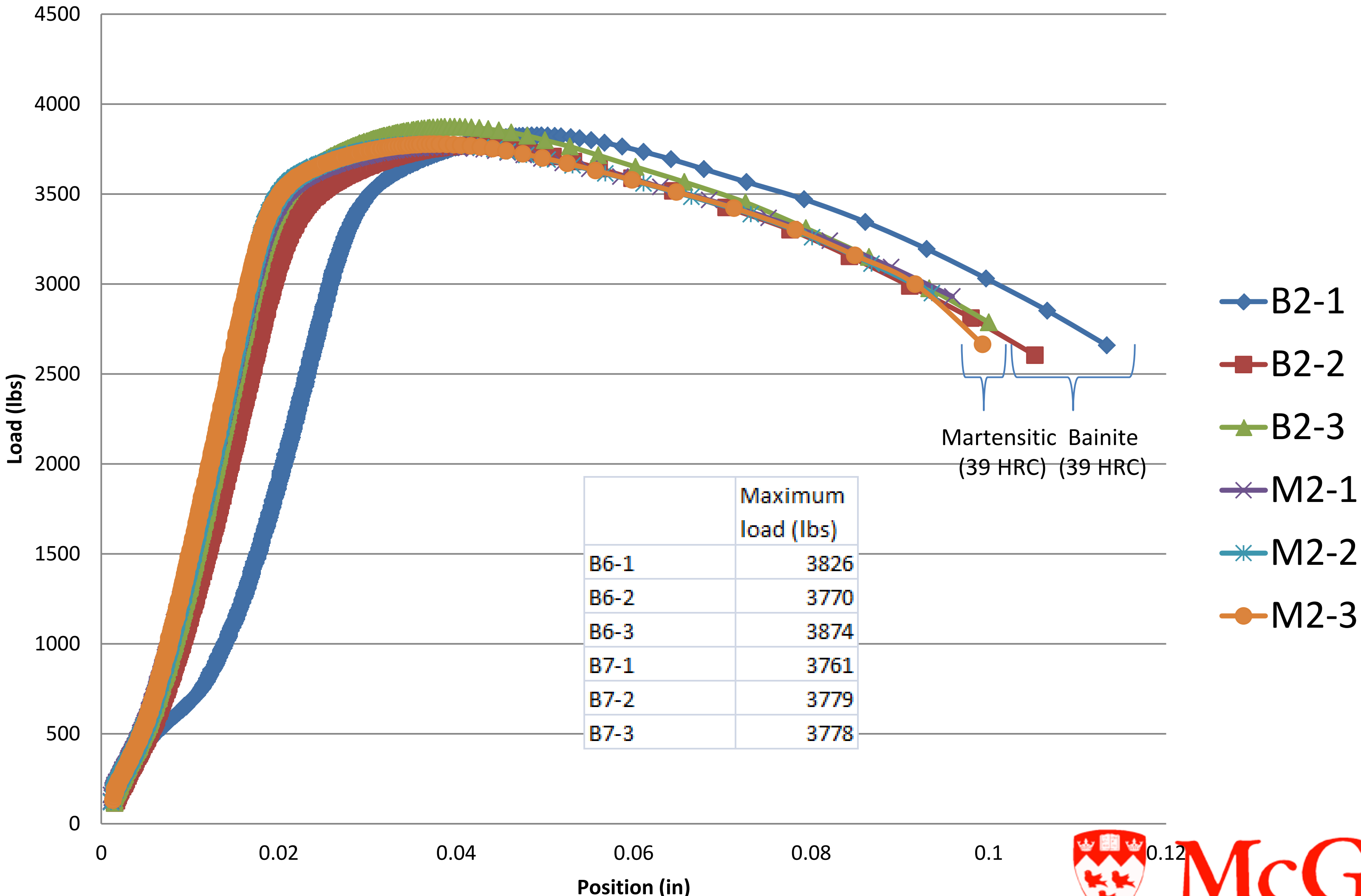
Sample condition- Tensile

B6 (Bainite) vs B7 (Martensite) Tensile Evaluation



	Maximum load (lbs)
B6-1	3826
B6-2	3770
B6-3	3874
B7-1	3761
B7-2	3779
B7-3	3778

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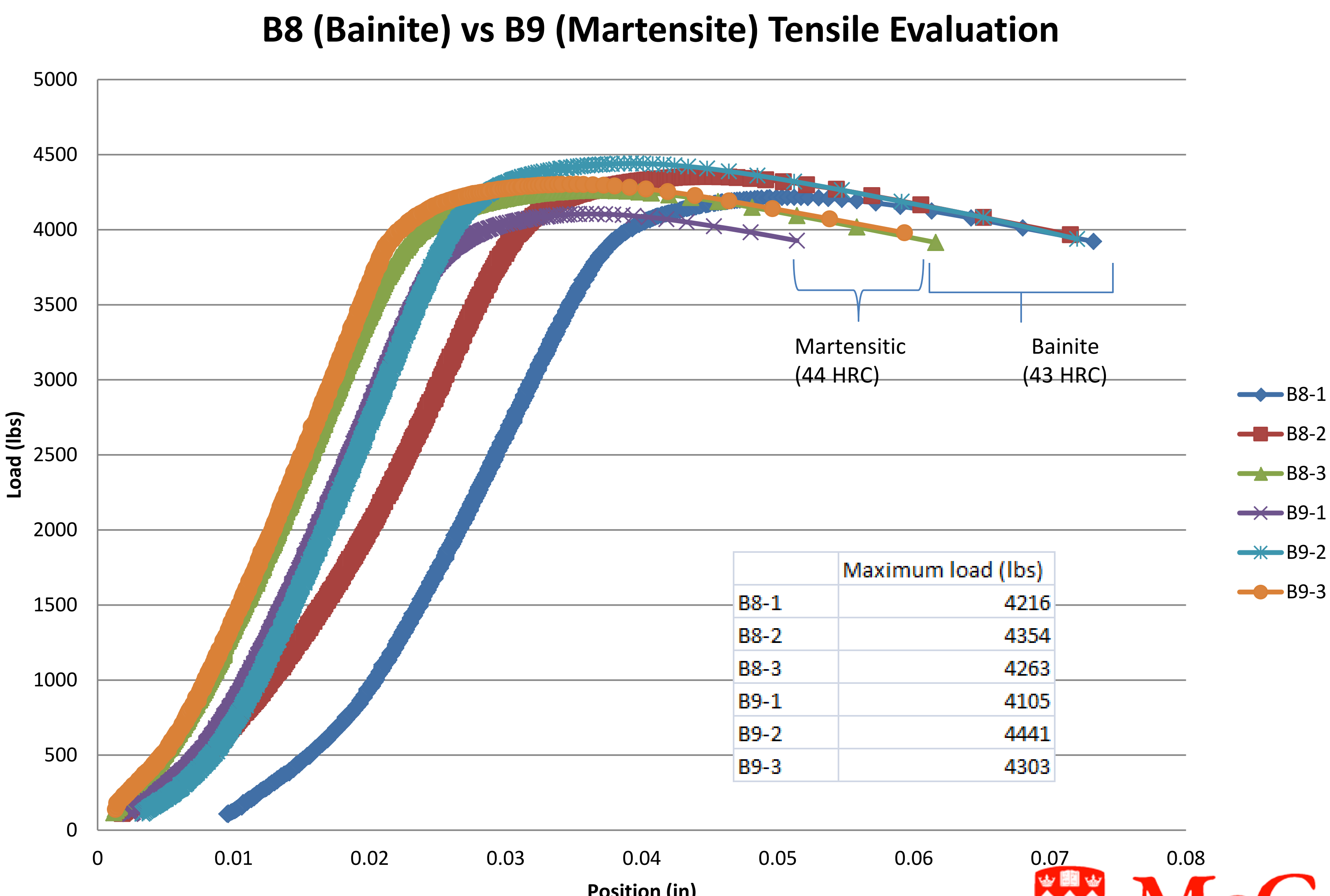
Sample condition- Tensile



	Maximum load (lbs)
B8-1	4216
B8-2	4354
B8-3	4263
B9-1	4105
B9-2	4441
B9-3	4303

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Sample condition- Tensile

	Tensile strength ksi	% Elong	% RA	HRC
B3 - Martensite	220.6	12.0%	21.2%	45
B4 - Bainite	226.5	19.4%	27.4%	47
B6 - Bainite	187.8	18.9%	37.4%	39
B7 - Martensite	184.6	19.0%	33.1%	39
B8 - Bainite	208.8	13.7%	31.8%	44
B9 - Martensite	211.2	17.0%	27.8%	44

Note 5: Tensile test results showed higher ductility for the bainitic samples.
Note 6: tensile specimens were machined and, i.e., removed effect of work-hardening from rolling after HT for samples B6, B7, B8 and B9.

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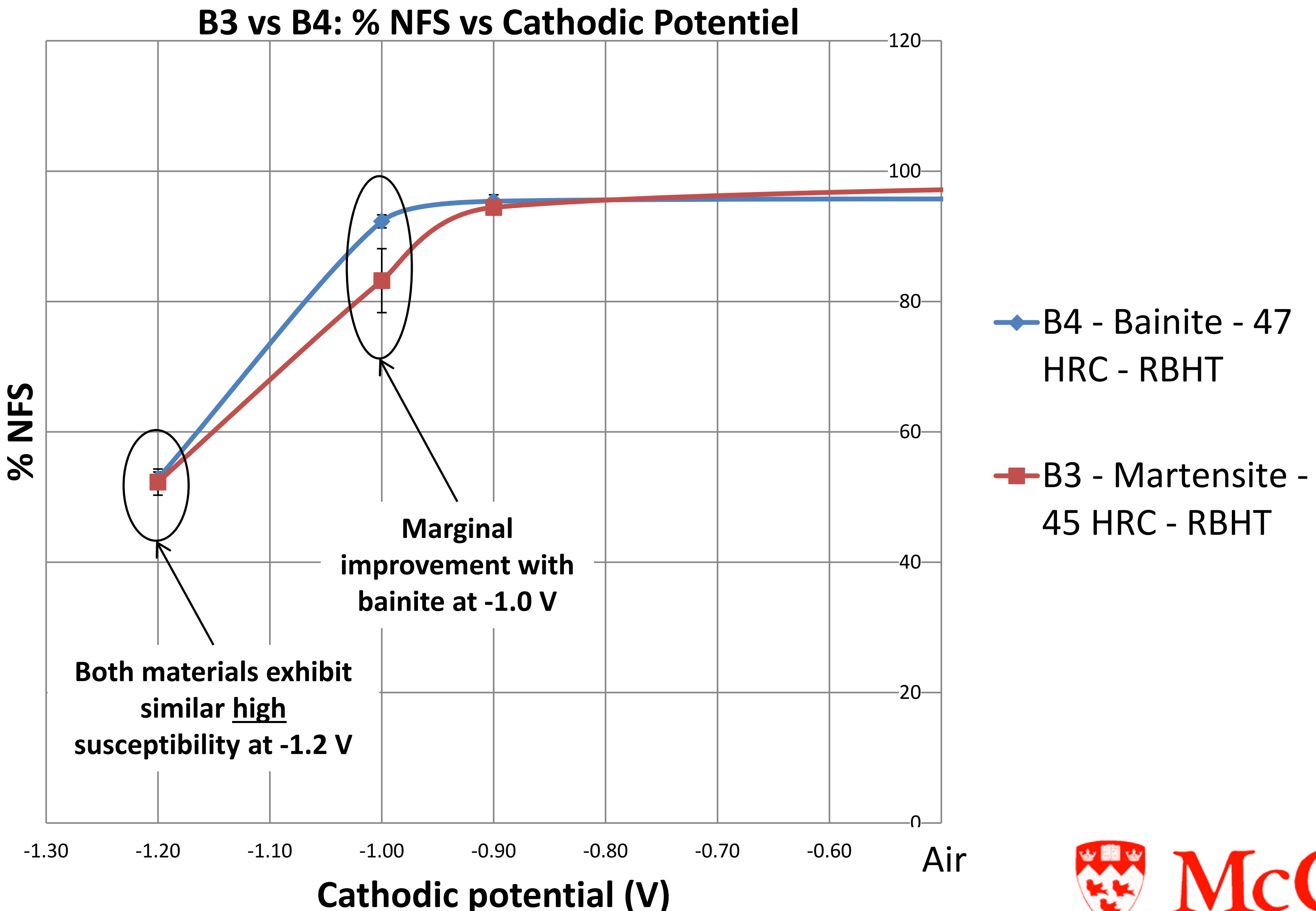
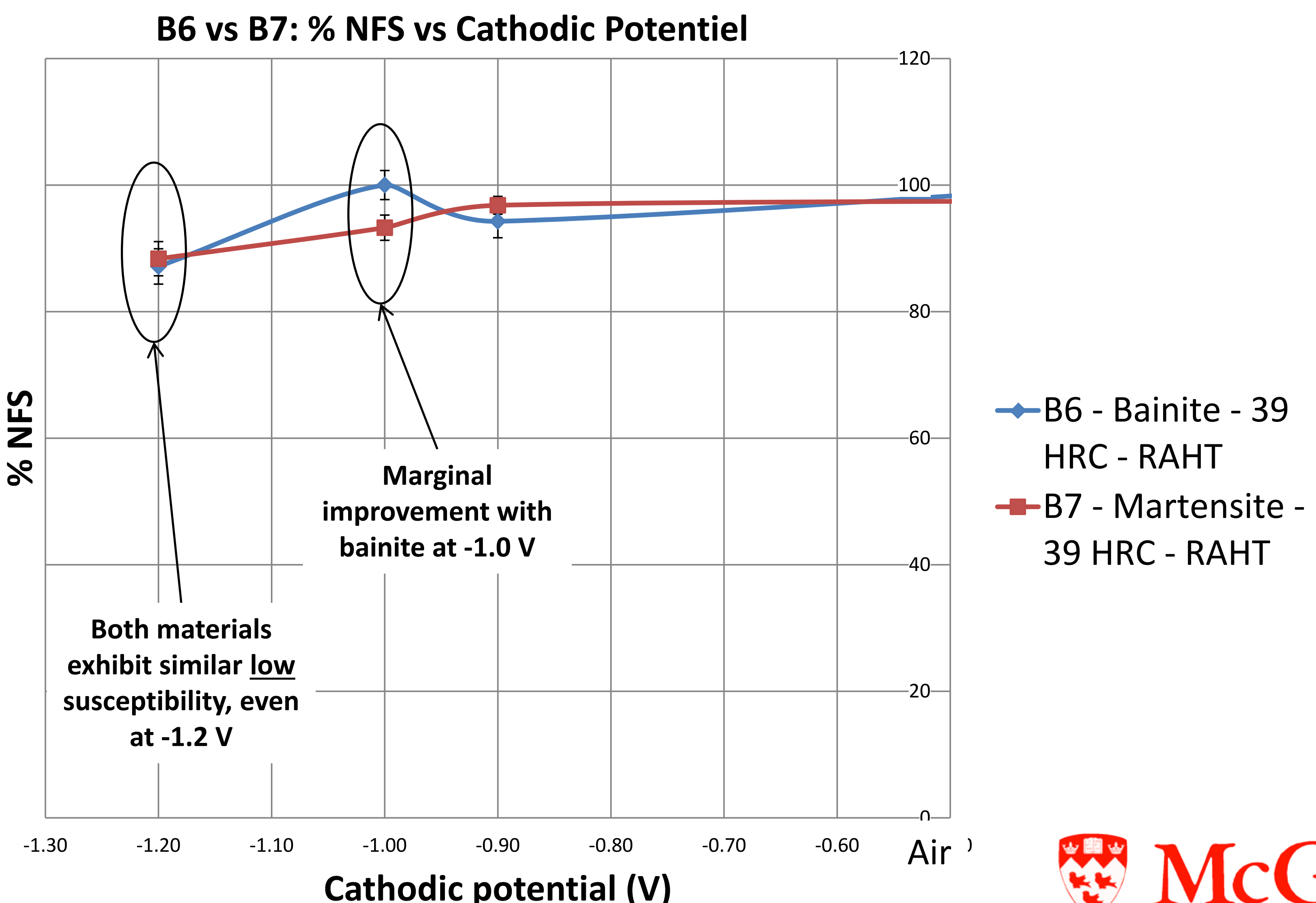
% NFS - Rolled before HT ~45 HRC



% NFS - Rolled after HT ~ 39 HRC



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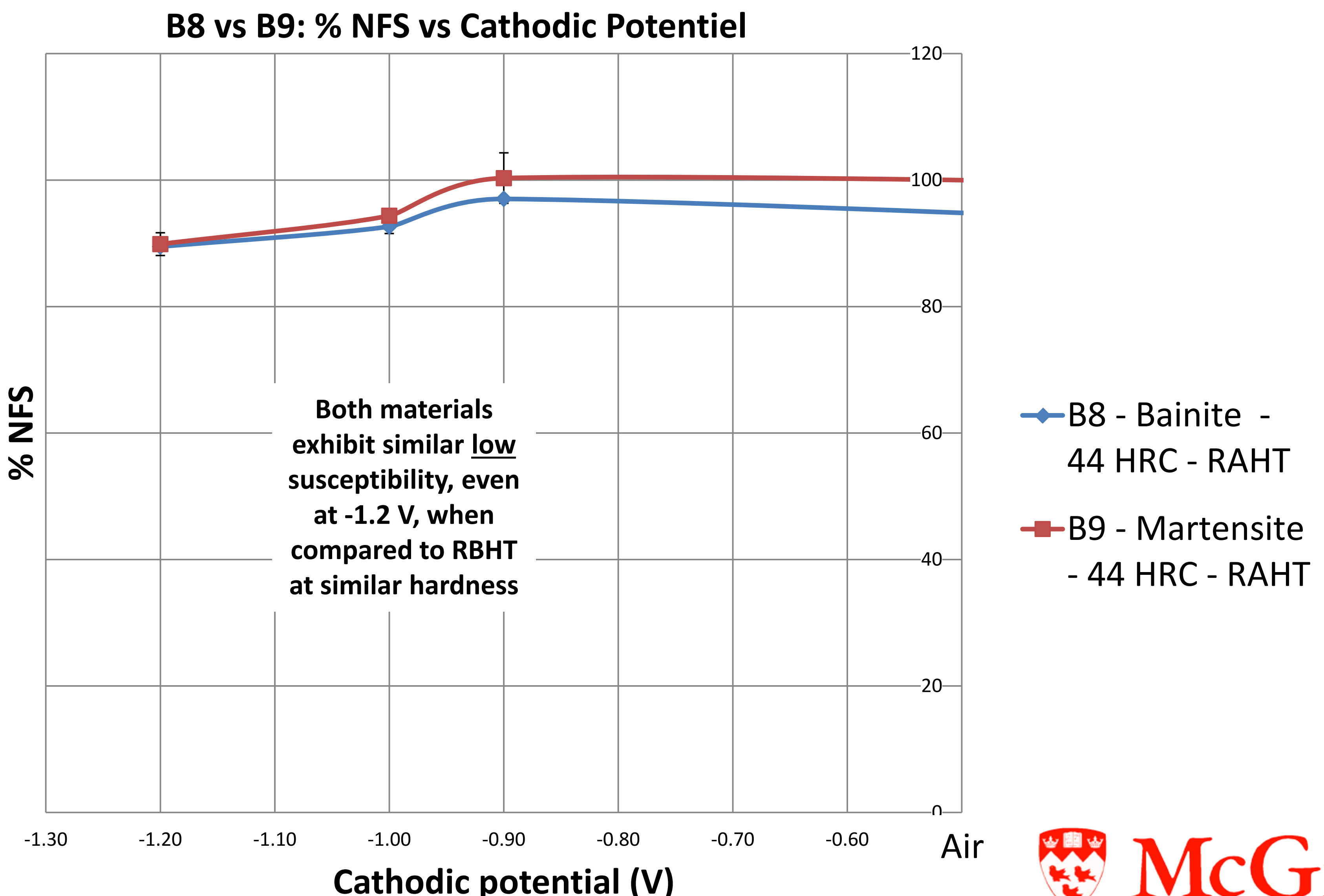
% NFS – Rolled after HT ~ 44 HRC



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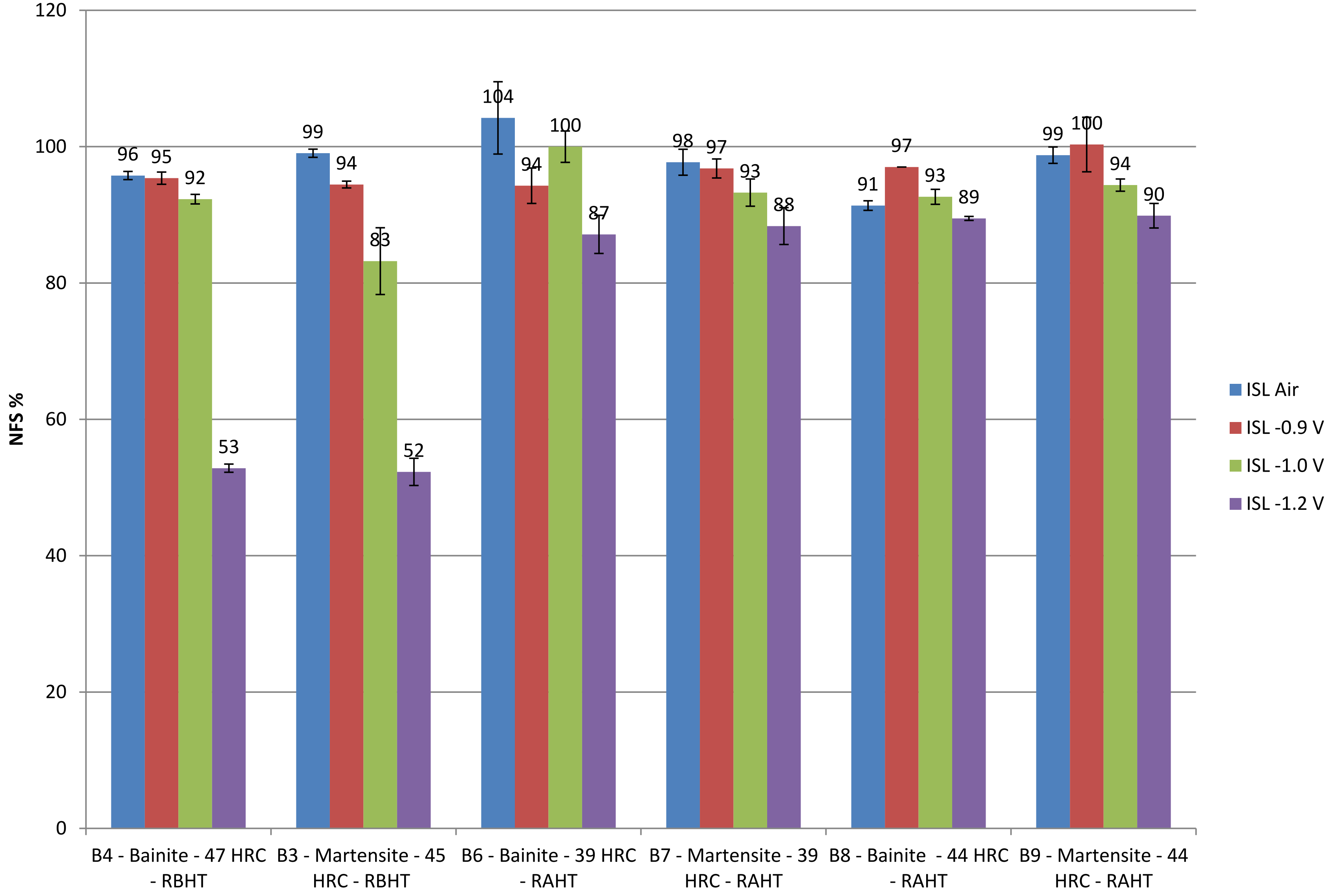
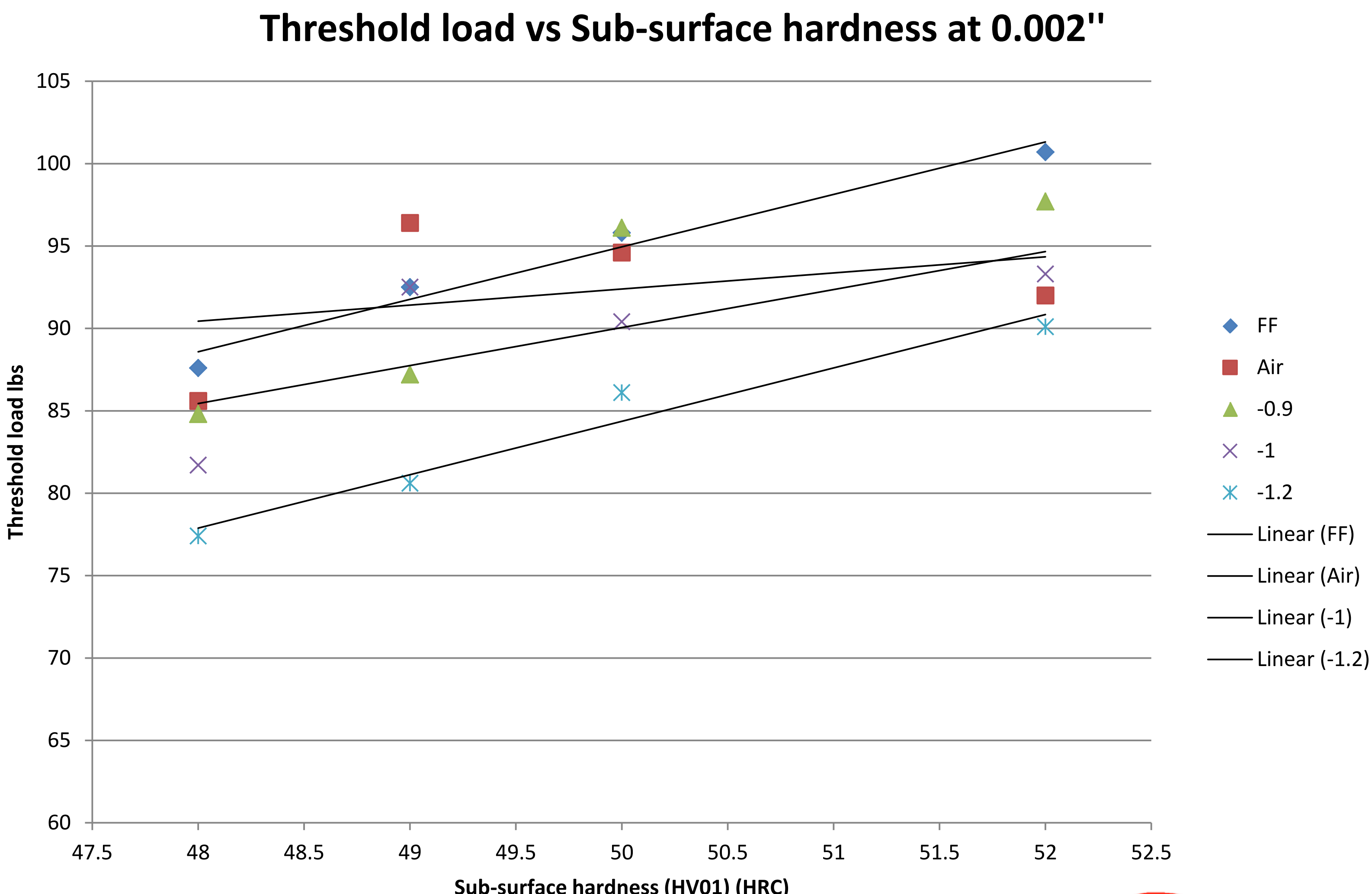
Results – ISL – % NFS



Note 7: ISL testing results showed similar NFS% between martensitic and bainitic samples – independent of hardness.

Note 8: Rolling after heat treatment had a marked effect of reducing HE susceptibility. NFS % losses were between 10-13% vs. close to 50% for rolled before heat treatment. These results can be explained by an increase in dislocation density during rolling after heat treatment.

Results – Dislocation density effect



Note 9: Dislocations are H traps and can also block it from diffusing thru the matrix. It is observed as sub-surface hardness increases, threshold load also increases. The result can be explained by the effect of increased dislocation.

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