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Hardness Humps or Decarburization? Misclassification of Subsurface Microhardness Anomalies in Vacuum-Oil-Quenched Steel

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***Abstract:** Surface-to-core microhardness variations in vacuum-oil-quenched 300M ultra-high-strength steel frequently trigger rejection under industrial standards such as AMS 2759, which interpret hardness traverse deviations as evidence of decarburization or carburization. This study demonstrates that such rejections often reflect a fundamentally different set of mechanisms: transient furnace atmosphere effects, microstructural banding, and measurement geometry bias rather than carbon chemistry change. Twenty-five controlled specimens were evaluated using microhardness traverses, metallography, EDS, and Residual Gas Analysis. Results show that failing profiles exhibit a non-monotonic hump or dip morphology physically incompatible with diffusion-controlled carbon transport, that no measurable carbon gradient exists at the surface of rejected specimens, and that coated specimens processed identically to failing uncoated specimens consistently pass the hardness criterion. Microstructural banding alone produces up to 70 HK scatter between adjacent indents independent of surface chemistry. Current industrial standards do not distinguish between profile shapes consistent with carbon gradients and those driven by quench-microstructure effects, leading to systematic misclassification of metallurgically sound parts. A structured supplementary evaluation framework is proposed.*

***Keywords:** 300M steel, vacuum oil quenching, decarburization, AMS 2759, microhardness traverse, residual gas analysis, microstructural banding, quench wetting.*

I. INTRODUCTION

300M low-alloy ultra-high-strength steel is widely used in aerospace structural applications, including landing gear and airframe fittings, where tensile strengths exceeding 280 ksi are required. Industrial acceptance of heat-treated components is governed by specifications including AMS 2759/2 and Boeing BAC 5617, which assess surface integrity through microhardness traverse criteria. These criteria define the allowable hardness variation from surface to core, typically no more than 25 Knoop units on the assumption that deviations indicate changes in carbon chemistry: decarburization producing a soft surface zone, or carburization producing a hard one.

This assumption is physically well-founded for atmosphere furnace processing. It is significantly less appropriate, and this paper argues sometimes incorrect, when applied to high-vacuum oil quenching of high-hardenability steels. In practice, vacuum-oil-quenched 300M parts regularly exhibit subsurface hardness anomalies localized humps or dips at 0.004–0.025 inches depth and are rejected under decarburization criteria despite having no measurable surface carbon change and no microstructural decarburization features. These parts may be fully hardened and metallurgically sound in every respect except the hardness traverse result. The economic and quality consequences of this misclassification are significant. This paper identifies the actual mechanisms responsible, demonstrates that current standards do not capture them, and proposes a revised evaluation approach.

II. BACKGROUND AND THEORETICAL FRAMEWORK

A. Classical Carburization and Decarburization

Carburization and decarburization are governed by Fick's second law of diffusion. For a surface exposed to a carbon potential different from the bulk steel, the resulting concentration profile is:

$$\mathbf{C(x,t) = C_s + (C_0 - C_s) \cdot erf(\, x / 2\sqrt{Dt}\,)}$$

where x is depth, t is time, D is carbon diffusivity in austenite ($\sim 10^{-11}$ m²/s at 1600 °F). This solution always produces a monotonically varying concentration profile — and therefore a monotonically varying hardness profile after quenching. A hardness profile that rises then falls, or falls then rises, cannot under any physically realistic boundary condition be the product of diffusion-controlled carbon transport. This geometric constraint is the foundational diagnostic tool of this study.

B. The Limitation of Industrial Standards

AMS 2759 applies its hardness traverse criterion uniformly to all failing profiles without requiring assessment of profile shape, metallographic examination for free ferrite, or atmosphere data review before a rejection is finalized. A non-monotonic profile — physically inconsistent with carbon diffusion receives the same rejection outcome as a monotonic profile that genuinely indicates decarburization. This structural limitation is the source of the misclassification problem this paper addresses. The standard was developed for atmosphere furnace environments where carburization and decarburization are genuine, common failure modes. Applied to vacuum oil quenching, where the physical mechanisms governing surface condition are fundamentally different, it produces a systematic false-positive rejection rate for a specific class of hardness anomaly.

III. EXPERIMENTAL METHODOLOGY

Specimens of 300M bar stock were austenitized at 1600 °F under approximately 50-micron vacuum with soak times of 10 and 240 minutes, then oil or water quenched under varying agitation conditions. Twenty-five specimens covering full-diameter and quarter-section geometries, chordal and non-chordal traverse methods, and coated and uncoated surface conditions were systematically evaluated. AISI 12L14 low-carbon steel sentinel specimens were co-processed to detect any furnace carburization tendency. Six additional specimens, two coated and four uncoated, were processed in a furnace previously associated with elevated non-conformance rates to isolate the effect of surface condition.

Characterization methods included: 500 gf Knoop microhardness traverses beginning at 0.002 inches from the prepared surface; optical metallography with 2% nital etch examining surface and core microstructure; EDS line scans from surface to 500 µm depth on raw, passing, and rejected specimens; and continuous Residual Gas Analysis (RGA) monitoring throughout the heat treatment cycle.

IV. RESULTS AND DISCUSSION

A. Profile Shape: The Primary Diagnostic

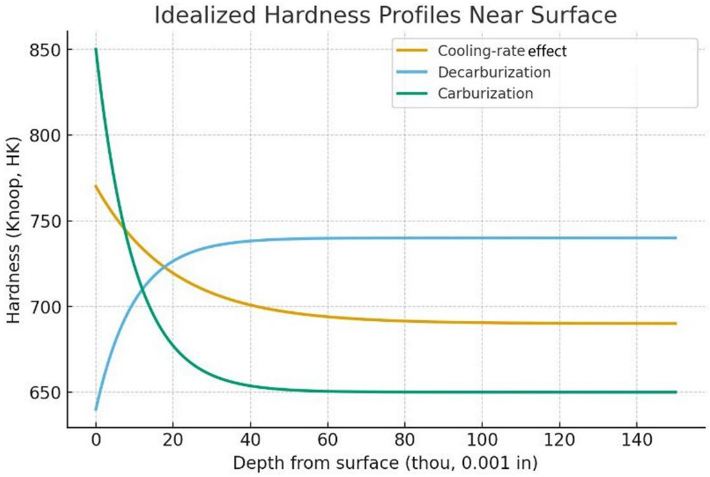

Figure 1: Typical carburization/decarburization hardness versus depth profile

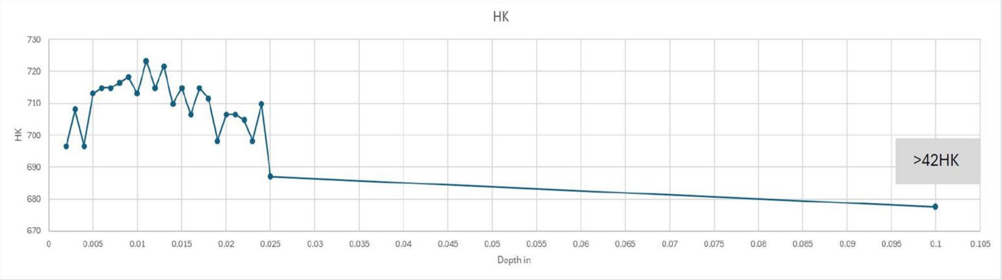

Figure 2 Hardness profile of a rejected 300M sample after vacuum oil quench showing a subsurface hardness anomaly (hump) that does not match a classic carburizing or decarburizing profile.

Rejected production specimens consistently exhibited non-monotonic hardness profiles: surface hardness within specification, a localized hump or dip at 0.004–0.025 inches depth reaching deviations of up to 70 HK, and core hardness returning to a value similar to the surface. This profile shape is geometrically incompatible with Fick's law diffusion kinetics under any boundary condition. A carbon gradient cannot produce a hardness peak that resolves back to a baseline value at depth. The non-monotonic shape alone is sufficient to redirect investigation away from carbon chemistry and toward heat-transfer and microstructural mechanisms, a conclusion derivable directly from the traverse data, requiring no chemical analysis.

Measurement depth sensitivity: The first microhardness indent at 0.002 inches drove 87% of all non-conforming results. The failing rate declined substantially by 0.006 inches, localizing the phenomenon to the extreme near-surface zone most sensitive to surface condition and quench wetting behaviour. The chordal traverse method produced an 88% failing rate versus 53% for non-chordal traverses on matched specimens, and quarter-section cut specimens failed at 80% versus 53% for full-diameter specimens.

B. Furnace Atmosphere and Ultra-Thin Oxide Formation

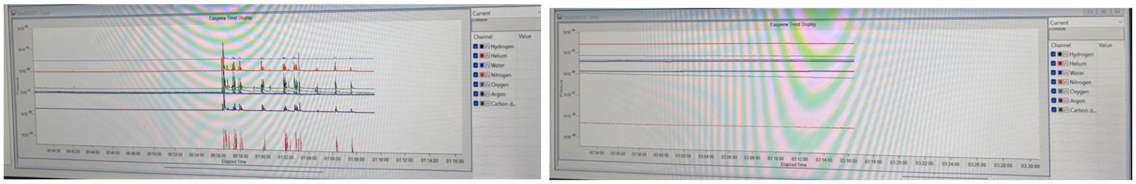

Figure 3 RGA atmosphere traces during production heat treatment cycles: (left) before bake-out, showing intermittent multi-gas spikes during heat-up; (right) after cleaning and bake-out, showing substantially reduced spike activity.

RGA monitoring revealed intermittent spikes in H_2O, CO_2, O_2, and H_2 during the heat-up phase between 400 and 1200 °F, invisible to bulk pressure gauging but representing genuine transient increases in local oxygen activity at the steel surface. These spikes originate from outgassing of hot-zone insulation, graphite elements, fixtures, and the load itself. Even brief exposure to elevated oxidizing species at austenitizing temperature is sufficient to form nanometre-scale surface oxide films that, while undetectable by conventional metallography, alter the wetting behaviour of quench oil against the steel surface.

Oil quench heat transfer in the vapour-blanket stage is critically sensitive to surface condition: a surface oxide shifts the vapour-blanket breakdown temperature and locally modifies heat extraction rate in the extreme near-surface zone precisely where the hardness anomalies appear. Following furnace cleaning and bake-out, RGA spike activity decreased substantially, and hardness non-conformance rates improved correspondingly. Two distinct hot-zone contamination regimes were identified:

- Graphite-attack regime (Furnace 1 elements): persistent low-level oxidizing activity progressively gasified graphite through $C + H_2O \rightarrow CO + H_2$ and $C + CO_2 \rightarrow 2CO$ reactions, producing pitted, porous element surfaces visible on inspection.
- Condensate regime (Furnace 2 elements): transient high-intensity outgassing drove metal vapour transport and deposition of Fe-, Mn-, and Cr-bearing films on element surfaces, with concurrent micro-scale surface oxide formation on parts at austenitizing temperature.

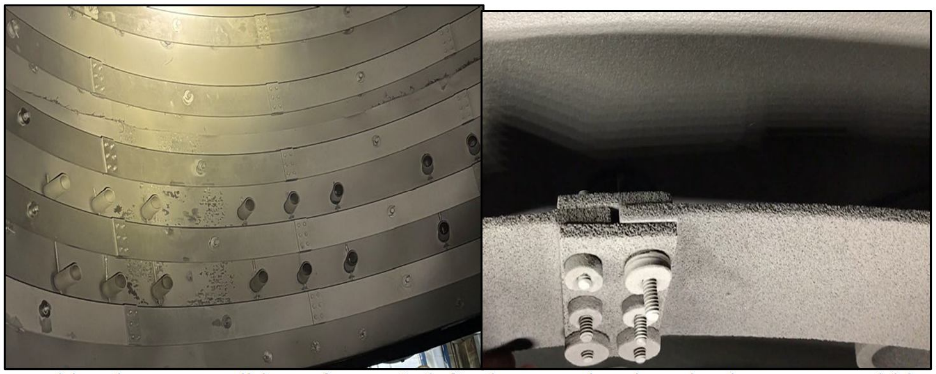

Figure 4 Hot-zone graphite element conditions: furnace1 (left) elements showing pitted, porous, sand-blasted texture from progressive oxidative gasification; furnace2 (right) elements showing smooth substrate with irregular metallic/oxide condensate patches.

C. Microstructural Banding and Chemical Analysis

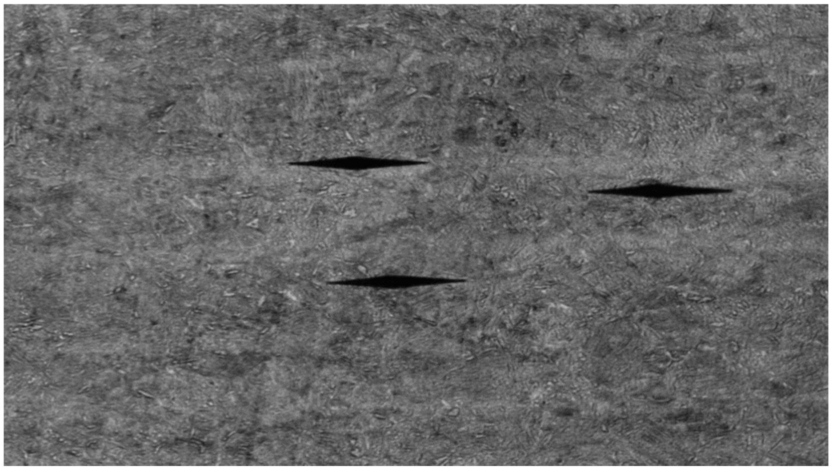

Figure 5 Optical micrograph of core region in 300M showing banded martensitic microstructure. Three adjacent indents at nominally identical positions returned 671, 693, and 629 HK — a 70 HK spread from banding alone, exceeding the 25 HK specification limit by a

Optical metallography at the core of 300M specimens revealed pronounced microstructural banding alternating regions of differing martensite morphology and retained austenite content from Mn, Ni, Cr, and Mo microsegregation during ingot solidification. Three adjacent indents at nominally identical core positions returned 671, 693, and 629 HK: a 64 HK range and a 70 HK difference between two adjacent readings. All three fell within acceptable individual core hardness limits, yet the traverse variation between them exceeded the specification limit by a factor approaching three. This banding-induced scatter is inherent to the material and cannot be eliminated by process optimization.

EDS and metallographic findings: EDS line scans on raw, passing, and rejected 300M specimens showed no measurable difference in surface carbon signal between any condition. No directional trend, no enrichment, and no depletion was detectable in any rejected specimen. Metallographic examination found no free ferrite, no decarburized surface layer, and no carburized case in any specimen. All microstructures were consistent with fully hardened 300M retained austenite, lath martensite, and coarse martensite. The 12L14 sentinel steel showed no carburization after co-processing, confirming the furnace atmosphere was not capable of measurable carbon enrichment under tested conditions.

V. WHY INDUSTRIAL STANDARDS DO NOT CAPTURE THIS PHENOMENON

The evidence assembled in this study supports a specific and practically important argument: the hardness traverse criterion in AMS 2759, as currently applied to vacuum oil quench processing, does not distinguish between two physically distinct failure modes that produce outwardly similar test results but have completely different causes, different implications for part integrity, and different appropriate corrective actions.

True carburization or decarburization involves a genuine change in surface carbon content, produces a monotonic hardness profile consistent with Fick’s law, and is accompanied by metallographic features including free ferrite or a carburized case.

The quench-microstructure phenomenon described in this paper involves no change in surface carbon content, produces a non-monotonic hardness profile inconsistent with any diffusion model, has no accompanying metallographic decarburization features, and is driven by transient atmosphere effects on quench wetting combined with material banding. Rejecting parts on this basis misidentifies the cause and misdirects corrective action.

AMS 2759 does not require profile shape assessment before a rejection decision. It does not require metallographic confirmation of free ferrite. It does not require RGA data review. It applies one criterion uniformly across both failure modes and both processing environments, atmosphere furnace and vacuum furnace despite the fact that the physical mechanisms operating in these environments are fundamentally different. This is not a flaw in the standard for its intended application domain; it is an application of the standard outside the physical envelope it was designed to cover, producing a systematic misclassification rate for vacuum-oil-quenched high-hardenability steels.

VI. PROPOSED REVISED EVALUATION FRAMEWORK

When a vacuum-oil-quenched part fails the hardness traverse criterion, the following supplementary steps are proposed before final disposition. Any alternate acceptance decision should be subject to appropriate engineering authority review.

1) Assess profile shape. If monotonic, standard rejection applies. If non-monotonic with surface and core within specification, proceed to Step 2.
2) Examine metallography for free ferrite. Absence of free ferrite and decarburization microstructure in a failing specimen is strong evidence against a carbon chemistry cause.
3) Review RGA data for the specific furnace run. A clean RGA trace supports a non-chemical origin. Significant spike activity warrants bake-out and hot-zone inspection before further production.
4) Perform quantitative carbon analysis where resources permit, using EPMA/WDS or LECO combustion analysis. Absence of a carbon gradient confirms non-chemical origin.
5) Assess for microstructural banding at the depth of deviation. If prominent banding is present and consistent with the deviation location, document and consider additional traverses at alternate circumferential positions.
6) Implement furnace corrective action regardless of part disposition. Bake-out and hot-zone inspection address the root cause of transient atmosphere events for subsequent production.

VII. CONCLUSIONS

Subsurface microhardness anomalies in vacuum-oil-quenched 300M steel are driven by a combination of quench wetting perturbation from transient furnace atmosphere oxide formation, microstructural banding from steelmaking segregation, and measurement geometry sensitivity not by carburization or decarburization. The key conclusions of this study are:

1) The non-monotonic profile shape of failing 300M specimens is geometrically incompatible with diffusion-controlled carbon transport under any boundary condition, providing direct evidence against a carburization or decarburization mechanism independent of chemical analysis.
2) EDS detected no measurable carbon gradient at the surface of any rejected specimen. Metallographic examination found no free ferrite in any specimen. All microstructures were consistent with fully hardened 300M.
3) RGA monitoring revealed transient gas spikes invisible to bulk pressure gauging that are capable of forming surface oxide films sufficient to alter oil-quench wetting and produce near-surface hardness deviations through a thermal mechanism, not a chemical one.
4) Coated specimens consistently passed the hardness criterion in the same furnace and cycle that produced non- conforming uncoated specimens, directly isolating surface condition at quench entry as the governing variable.
5) Microstructural banding produced hardness differences of up to 70 HK between adjacent indents at nominally identical positions, representing a second source of hardness scatter inherent to the material and independent of process chemistry.
6) Industrial standards including AMS 2759 do not distinguish between monotonic and non-monotonic failing profiles and do not require profile shape assessment, metallographic confirmation, or RGA review before rejection is finalized, leading to systematic misclassification of metallurgically sound parts processed by vacuum oil quenching.

A guideline separating true chemical decarburization/carburization from microstructural subsurface humps is necessary to avoid misclassification of acceptable parts as non-conforming and to ensure corrective actions are appropriately targeted.

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