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PROCESSING INFORMATION

Processing Information

Manufacturing and Thermal Processing of *Ferrium*® C64™

This document will be periodically updated by QuesTek, as additional data is generated for this new material. Please contact QuesTek to ensure that you are using the most current version of this document.

1. Purpose:
   a. This document provides introductory information regarding manufacturing and thermal processing of components made from QuesTek-designed *Ferrium* C64 steel. This information is intended to assist purchasers, fabricators and users in their application engineering of C64 to design and produce specific components or pieces; those individuals and companies remain wholly responsible for the outcome of their decisions and processes. This document does not supersede applicable Reference Documents and Standards such as those listed below.
2. Reference Documents and Standards:
   a. UNS K92731
   b. SAE AMS 6509
   c. SAE AMS 2759/7 for thermal processing
   d. SAE AMS 2759/9 for hydrogen bake out parameters
   e. SAE AMS 2759/11 for stress relief parameters
3. Summary:

   C64 is a new high-strength, good-toughness, high-hardenability, secondary-hardening steel that is produced by vacuum induction melting followed by vacuum arc remelting (*i.e.,*VIM/VAR). When compared to steels such as AISI 9310 (“9310”) or EN36, some key processing differences to consider (described further below) include:
   a. Machining operations will differ; preliminary information is provided herein.
   b. Forging process parameters will differ, and a higher forging temperature will typically be required when forging C64 in an existing die previously used for forging 9310.
   c. Using low pressure (vacuum) carburization rather than conventional gas carburization is highly recommended. Various carburization cycles with varied hardness profiles for C64 have been and are being developed by various heat treating vendors, and using these vendors may be most expedient (especially for prototype production). Contact QuesTek for more information about these vendors.
   d. It is highly recommended that areas adjacent to carburized surfaces (*e.g.* the tips and side faces of gear teeth, splines, etc.) be masked with copper plating, stop-off paint, or other suitable means in order to prevent excess carburization at the edges/corners of carburized surfaces.
   e. The high carburization temperature of C64 (1830°F or 999°C) can increase the propensity of parts to sag during processing, so it is important to adequately support parts (especially long horizontal sections), or hang parts vertically as feasible.
   f. The tempering directions listed below should be closely adhered to. Because C64 is a high hardenability steel, it achieves full martensitic transformation even at slow quench rates. Therefore, a single tempering step is sufficient to achieve full mechanical properties. Over-tempering of C64 may result in lower-strength / higher-toughness properties. Rapidly quenching from the tempering temperature may result in higher strength, lower toughness, and more distortion.
   g. Operations such as shaft straightening (if required) should preferably be done after carburization but prior to the temper. C64 achieves full mechanical strength after tempering, and thus trying to straighten after tempering will be more difficult.
   h. The high hardenability of C64 allows the use of slower/milder quench rate which can reduce total distortion, including in large parts. When vacuum carburizing C64, it may be possible to replace final grinding operations with lapping operations to reduce costs and schedules.
   i. In the case of mating gears of different materials (e.g., a 9310 ring gear mating with a C64 pinion), special consideration should be given to the surface finish, and it is recommended that both surfaces be finished to similar finish and roughness. A rougher surface finish, especially on the harder C64, may cause pitting on softer 9310 material.

j. Shot peening and isotropic superfinishing processes will differ; preliminary information is provided herein.

4. <u>Forging</u>

a. Please contact QuesTek to discuss your C64 forging application especially when forging from ingot stock input. Best practices for forging have not yet been fully established for C64, but forging of *Ferrium* C61 (an alloy closely related to C64) has been done routinely and successfully.

b. On a preliminary basis, the suggested procedure for forging C64 parts from <u>billet and bar</u> stock follows (if higher hot-fire temperatures are preferred, then use the procedure in part "c" below):

i. Hot fire at approximately 1800°F - 2050°F (982°C - 1121°C), with no requirement on forging reduction ratio.

ii. Cool to room temperature after forging operations are complete.

iii. Normalize at 1785 to 1875°F (974 to 1024°C) for 1 hour and air cool to room temperature. Using the higher end of this temperature range may increase grain size (leading to decreased strength and toughness) but provide even further reduction in distortion during carburization; using the lower end of this range will have the opposite effect.

iv. Anneal at 1250°F (677°C) for not less than 2 hours and air cool to room temperature.

v. The hardness should be less than 327 HBW.

c. On a preliminary basis, the suggested procedure for forging C64 parts from billet and bar stock at higher hot-fire temperatures, or from <u>ingot stock that has been fully homogenized</u>, follows:

i. Hot fire at approximately 2300°F (1260°C), with a minimum of 4:1 forging reduction ratio. The majority of the 4:1 reduction should be completed in one final pass in order to meet grain size requirements.

ii. Cool to room temperature after forging operations are complete.

iii. Normalize at 1785 to 1875°F (974 to 1024°C) for 1 hour and air cool to room temperature. Using the higher end of this temperature range may increase grain size (leading to decreased strength and toughness) but provide even further reduction in distortion during carburization; using the lower end of this range will have the opposite effect.

iv. Anneal at 1250°F (677°C) for not less than 2 hours and air cool to room temperature.

v. The hardness should be less than 327 HBW.

vi. Confirm that mechanical properties (UTS, YS, elongation, RA, K_{IC}) have been achieved on representative sample material after being fully heat treated.

d. Avoid hot-fire temperatures between 2150°F (1177°C) and 2250°F (1232°C), in order to avoid coarsening of the grain pinning dispersion particle, which may lead to grain growth.

e. Forging dies used for 9310 can probably be re-used with C61, although it will probably be necessary to forge C61 at higher temperatures than 9310. It is recommended to perform a trial forging after reviewing the available flow stress data. Contact QuesTek for more information.

5. <u>Cold Working</u>

a. There have been no prior attempts to cold-forge C64. Given the higher alloying content of C64 vs. 9310, a greater number of blows or greater forces are expected. The very high ductility and toughness of the C64 core suggests it should be capable of cold working. A simple, short trial run to make a few parts using C64 would probably yield the best information, preferably with the use of an older die nearing retirement in the unlikely event that any die damage occurs. For more information, please contact QuesTek.

6. <u>Machining</u>

a. C64 bar stock will typically be shipped from the mill or supplier in a normalized-and-annealed condition, with hardness of approximately 33-37 HRC, and with the decarburization later and surface defects removed. With respect to material removal rates, the behavior of C64 during grinding will be roughly comparable to 9310, but C64 will typically be more difficult to hob. It is generally difficult to grinding burn damage on C64 due to its high tempering temperature; users may want to consider using higher grinding speeds than for 9310, to improve machining times. Turning and hobbing operations are typically 30 to 50% longer for C64 compared to 9310, depending on the given application.

b. Optimization of milling and drilling operations for C64 is underway, and only preliminary guidance is presently available. Milling operations might begin by selecting a tool that is typically used for 400 series stainless steel and ~260 SFM and ~0.0025 IPT cutting speeds (actual chip load relative to radial engagement of tool), with coolant through the drill, and then adjust operations from there. Drilling operations might begin by selecting a drill bit that is typically used for 400 series stainless steel and ~250 SFM and ~0.005 IPR cutting speeds, with coolant through the drill, and adjust operations from there.

c. A related ITAR-distribution-restricted report from the National Center for Defense Manufacturing and Machining (NCDMM; www.ncdmm.org) dated Jan. 22, 2010 entitled "Manufacturing Evaluation of Innovation & Advanced Materials. Latrobe Specialty Steel – C61 & S53 Materials" is available. This report provides some initial machining analysis of *Ferrium* C61, which should machine very similarly to C64. Contact QuesTek for details.

7. Welding and Brazing

a. We are not aware of any significant investigations of welding or brazing of C64 yet. On a preliminary basis, we suggest:

i. Making weld sticks from C64 material, then TIG weld, and then temper that part at 392°F (200°C) for 1 hour and then asses results.

ii. Practices typically used for 8620, 9310, 4320, etc should be applicable to C64

iii. If the joint is non-structural, then it may be possible to braze this joint using a Silicon Bronze filler rod.

8. Allowances for and Control of Heat Treat Growth / Runout / Distortion:

a. Due to the high carburization temperature of C64, it is important to adequately support and fixture parts (especially long horizontal parts) during heat treatment in order to minimize sagging or bending distortion.

b. Dimensional changes should be taken into account as the preferred manufacturing and machining paths for components are being determined.

i. The heat treat growth/permanent expansion is isotropic and should be ~0.0005"-0.001" per inch in the core. The amount of heat treat growth/permanent expansion in the case is under evaluation.

ii. One example of observed runout was demonstrated on pinions 1.5" diameter round and 12" long of Ferrium C61, an alloy similar to C61. When quenched using 2 bar nitrogen, the total runout was 0.005"; so in this example, the runout was <0.0005" per inch. The amount of runout will depend on: part geometry; the forces used and internal stresses induced during machining operations; quench rate; manner of part loading during heat treatment; and other factors. If a significant amount of material is removed, or if very large forces are used during machining operations, then residual stresses could increase the amount of runout. Preventative and correct methods to reduce residual stresses follow.

c. If it is desired to stress-relieve a machined part in the mill-annealed condition as a means to help prevent distortion during further processing (*e.g.* if significant forces were imposed on the part during machining), then either of two options are recommended:

i. Produce the part in a rough-machined state with adequate stock material (e.g. 1/8") on all surfaces so that oxide scale can be removed, and then stress-relieve the part at ~1200°F (649°C) for 2 hours, followed by cooling in air. Then complete all final machining operations prior to carburizing, in order to remove the oxide scale formed at 1200°F (649°C). This option will be the most effective option to stress-relieve parts.

ii. Produce the part in a fully- or nearly-fully-machined state prior to carburizing, and then stress-relieve the part at 700°F (371°C) for 2 hours or 525°F (274°C) for 4 hours, followed by cooling in air.

d. If it is anticipated that a straightening operation may be required to correct distortion caused by quenching, then the part's distortion will preferably be measured and any corrective steps taken after the carburization, quench and sub zero treatment steps have been completed, but before tempering:

i. If excessive distortion exists after the carburization, quench and sub-zero treatment, then it is recommended to heat the part to 392°F (200°C) in air for 1 hour, hot-straighten the part (temperature determined by amount of force required to straighten part; temperature should be maintained below 700°F (371°C) to avoid any tempering or decarburization; small oxide layer may form at this temperature), and allow the component to air cool. The full temper cycle described below must then be applied.

ii. NOTE: This is only a recommendation. If it is inconvenient to measure distortion and straighten a component before tempering, then users can investigate hot-straightening parts with greater force after tempering. Some C64 users have reported that they were able to hot-straighten C64 parts of modest size after tempering, after heating the parts to 392°F (200°F) in air for 1 hour (may increase the temperature to 700°F (371°C) to avoid any additional tempering or decarburization; however, a small oxide layer may form at this temperature).

9. Heat Treatment (Carburizing and Tempering)

a. General Information

i. We strongly recommend that C64 be carburized using a low pressure (vacuum) process since all of the carburization development work has been optimized for low pressure (vacuum) carburizing processes. C64 can in theory be gas carburized, but very little development work has been done to optimize the processing conditions for gas carburizing. In time QuesTek may develop some information for gas carburization. All of the information provided herein pertains to using low-pressure (vacuum) carburizing.

ii. The vacuum carburization cycle used must be optimized for C64 for the specific case depth profile desired. Existing carburization cycles that are currently used for 9310 or *Pyrowear*® 53 will typically produce too much carbon uptake if applied to C64, which will likely create unwanted primary carbides in the microstructure and soot on the surface.

iii. While C64 can in theory be induction-hardened, it is not recommended in part because the process would be very time-consuming and achievable surface hardness will be lower. All induction hardening process efforts on C64 would be experimental. To further discuss your interest in induction-hardening, please contact QuesTek.

b. Selection of Vendor:

i. Specific vacuum carburization cycles for C64 have been and are being developed by various heat treating vendors, and using these vendors may be preferred for expediency. Where possible/feasible, QuesTek recommends that these vendors be selected for the production of initial prototype parts since they often have well-developed process cycles and more experience to "dial in" a specific case depth as required for the specific application. Contact QuesTek for more information about these vendors.

ii. If instead a heat treating vendor is selected that does not have proven cycles for C64, then provided below in Section "f" is some publicly-available introductory information to help a vendor gain experience in carburizing C64 in order to produce a surface hardness of ~63 HRC, with a case depth of ~0.060" at ~52 HRC, and a core hardness of 47-50 HRC. Such vendors may need to perform experiments to optimize their equipment and process cycles for C64 prior to processing of parts.

c. Cleaning:

i. Immerse parts for a minimum of 15 minutes in alkaline solution or other approved degreasing method in order to remove oil, grease and other surface contaminants prior to heat treating.

d. Masking:

i. Like other carburizing-grade steels, C64 can be copper-plated prior to carburization (in accordance with ASTM 2418 or other standards), in order to protect or shield the surfaces that are not to be carburized. Suitable stop-off paints may also be used.

ii. It is highly recommended that areas adjacent to carburized surfaces (*e.g.* the tips and side faces of gear teeth, splines, etc.) be masked with copper plating, stop-off paint, or other suitable means in order to prevent excess carburization at the edges/corners of carburized surfaces.

iii. Some users have reported that using a sodium nitrate-based stripping agent to remove the copper plate generated surface soot on the parts, and the bath became discolored. Stripping of copper plate has also been successfully achieved using sodium cyanide-based stripping agents, where no surface soot or bath discoloration was observed.

e. Part Loading:

i. Load parts so that the surfaces to be carburized do not touch any other surfaces and in a manner to ensure adequate circulation of carburizing gas to all significant surfaces. Due to the high carburization temperature of C64, take extra care to load parts and provide generous amounts of support/fixturing (especially for long horizontal components) in order to minimize the sagging that can occur at high temperature. If possible, consider hanging shafts vertically. Work with the heat treat vendor to ensure they understand the importance of loading to reduce distortion.

ii. It is recommended that at least one carrier piece be included in the center of each load to validate the hardness and microstructure produced during thermal processing. If provided, additional test pieces should be placed in close proximity to the carrier piece.

f. Vacuum Carburizing and Quenching:

i. Prior to beginning carburization, it is recommended to hydrogen clean C64 parts for at least 3 minutes at 1830°F (999°C). One method of doing so that has been successfully applied to parts in the past is to flow hydrogen through the furnace during the heat-up cycle and shut off the flow of hydrogen for 3 minutes after the furnace reaches 1830°F (999°C). For specific questions regarding the preparation of C61 parts for carburization, please contact QuesTek.

ii. Use heating rates that ensure temperature uniformity of parts. If the furnace is pre-heated prior to beginning carburization, then it is recommended that the pre-heat temperature be no higher than approximately 567°F (315°C) in order to reduce the formation of coarse carbides.

iii. Vacuum carburize at 1830°F +/- 25°F (999°C +/- 14°C) for sufficient time to produce the required case depth and with sufficient gas flow to produce proper case microstructure and carbon gradation. There is a non-linear interaction of boost and diffuse steps that determine the final carbon profile. One example of a prior successful carburization cycle that was developed for C61 (an alloy closely related to C64) using a boost diffuse cycle path performed under 23% acetylene gas flow follows as an illustration, but it will need to be optimized for/by your specific heat treat vendor and their furnace:

1. 1 minute boost, followed by 20 minute diffusion
2. 1 minute boost, followed by 30 minute diffusion
3. 1 minute boost, followed by 40 minute diffusion
4. 0.75 minute boost, followed by 45 minute diffusion
5. 0.75 minute boost, followed by 50 minute diffusion
6. 0.75 minute boost, followed by 60 minute diffusion
7. 0.75 minute boost, followed by 90 minute diffusion

iv. Gas quench the parts in 2 bar inert gas (*i.e.,* nitrogen, argon, or helium) to below 150°F (66°C) for a minimum of 15 minutes. Increasing the gas quench pressure is permissible, but will likely increase distortion.

NOTE 1: The carburization cycle presented above yields a case depth of approximately 0.060" (refer to Figure 1). Other carburization cycles can generate other case depths and profiles. Contact QuesTek for further information.

NOTE 2: If it is desired to achieve the core properties of C64 without carburizing, this can be done by performing an austenitizing step at 1830°F +/- 25°F (999°C +/- 14°C) for 1 hour followed by quench as described above, and then continue processing as below.

g. Sub-Zero Treatment (Refrigeration):

i. Within 8 hours of quench, cool to -100°F (-73°C) or lower and hold for a minimum of one hour, followed by air warming to room temperature. When doing this step, moisture condensation should be considered; one example of a way to reduce condensation is to seal the parts in a bag with inert gas prior to cooling.

h. Cleaning:

i. A cleaning process might be considered after carburizing and before tempering to prevent staining, if the appearance of parts is important. Immerse parts for a minimum of 15 minutes in alkaline solution or other approved degreasing method. This cleaning step is not required and not cleaning the parts at this point will not alter the mechanical properties.

i. Tempering:

i. Vacuum Tempering (is recommended in order to eliminate need for applying and removing stop-off paint):

1. Evacuate chamber to a recommended level of 10^{-4} torr to achieve optimum results. Using 10^{-3} torr or other less severe vacuum levels may also yield acceptable results, but can result in a small amount of surface oxidation/decarburization.
2. Temper at 925ºF +/- 10°F (496°C +/- 6°C) for 8 hours +/- 2 hour.
3. Cool to room temperature or below, by slow gas circulation (at rate equivalent to air cool). DO NOT QUENCH.

NOTE: In order to ensure uniformity in heating of parts and temperature uniformity within a vacuum furnace, after evacuating the chamber to 10^{-4} torr, it may be necessary to backfill the chamber to 700 microns with an inert gas such as argon or nitrogen prior to tempering. Talk to your heat treat vendor about this.

ii. Air Tempering (is an acceptable alternative to vacuum tempering, but is not recommended since it typically requires additional operations; in addition, some color tinting of exposed metal will probably occur):

1. In order to prevent decarburization or oxidation on specific part surfaces, you should consider covering those surfaces with stop-off paint. The superficial oxidation that occurs on bare surfaces during air tempering is typically extremely shallow (< 0.0005").
2. Temper at 925ºF +/- 10°F (496°C +/- 6°C) holding for 8 hours +/- 2 hour.
3. Remove the parts from the furnace to cool (because the parts will likely over-temper if left in the furnace to cool).
4. Air cool. DO NOT QUENCH.
5. Remove any stop-off paint or copper plating.

j. Inspection of Carburization Results:

An example of inspection requirements based on the carburization process described above is shown here. Inspection criteria will need to be developed for the specific carburization process. Confirm that the carrier piece achieves these minimum property requirements:

i. Surface hardness greater than or equal to 62 HRC (69 HR45N or 91 HR15N).

ii. Case profile similar to Figure 1, meeting or exceeding the minimum hardness profile

1. 52 HRC case depth of 0.060", minimum 0.054"

iii. Acceptable microstructure (typical micrograph shown in Figure 2):

1. Should contain no networked carbides
2. Primary carbides should not exist below a depth of 0.0035"

iv. If test coupons are provided, mechanical testing should meet the minimum properties provided in SAE AMS 6509. Typical properties are shown in Table 1.

10. Final Machining and Warm Working

a. If necessary, perform final straightening or hot working of parts as outlined in Section 8.c and 8.d.

b. Vacuum carburization of C64 may provide the option to replace final grinding operations with a lapping operation (depending on the component and its application) due to the substantial elimination of intergranular oxides as compared to gas carburization. This potential savings in costs and schedule can be evaluated as component designs and manufacturing paths are being determined.

c. If final grinding is required, consideration should be given to the shape of the case profile to ensure that peak hardness will remain after grind stock removal.

d. In the case of mating gears of different materials (e.g., a 9310 ring gear mating with a C64 pinion), special consideration should be given to the surface finish, and it is recommended that both surfaces be finished to similar finish and roughness. A rougher surface finish, especially on the harder C64, may cause pitting on softer 9310 material.

11. Peening and Other Final Cold Working Processes

    a. Recommended shot peening parameters for C64 are currently being developed. Existing shot peening processes established for gear steels such as 9310 may not provide a significant mechanical benefit when applied to C64 parts (due in part to the much higher hardness of C64). Existing shot peening processes established for *Pyrowear*® 53 steel may be more appropriate to apply to C64 parts. Contact QuesTek for more information. Some success has been achieved shot peening with S110 shot, 0.008-0.012 A and with 200% coverage.

    b. Processes for laser peening and cavitation peening of C64 have been investigated. Contact QuesTek for more details.

12. Superfinishing

    a. Isotropic superfinishing (i.e. chemically-accelerated vibratory surface finishing) processes for C64 are currently being optimized. Existing isotropic superfinishing processes established for other gear steels such as 9310 have been observed to negatively affect surface finish/properties on C64. Superfinishing parameters used for C64 are different than those of 9310, and using parameters designed for 9310 may yield unsatisfactory, and potentially negative, results on C64. Contact QuesTek for more information about how to isotropically superfinish C64 components.

    b. Mechanically-induced superfinishing procedures (non-chemically-accelerated) are currently being evaluated for C64. Please contact QuesTek for more information.

13. Etching and Other Inspection:

    a. The specific etchant used on C64 will differ with the desired detail of observation. The following details have been observed successfully using the corresponding etchants:

        i. Grain size detail observations are optimized with the use of an oxidation etch.

        ii. Other microstructural detail observations (e.g. inclusions, martinistic substructures, primary carbides, lath packets, retained austenite and carbides, etc.) are optimized with the use of 2% Nital etch.

        iii. Macrostructural detail observations (e.g. temper/grinding burns, forging defects, grain pattern, etc.) are optimized with the use of 50% HCl in H_2O solution, etched at 160°F for 30 minutes.

    b. Measurement of residual stress in C64 components using x-ray diffraction techniques will typically require a greater number of calibration data points for the lattice spacing at various test angles than for 9310. Unlike 9310, the C64 curve is non-linear and behaves more similar to that of 52100. Contact QuesTek for details.

14. Coatings and Additional Final Processes

    a. C64 components can typically be coated or plated in similar manner to 9310 or other steels, with the added benefit that C64 has a high tempering temperature of 925°F (496°C), and may offer benefits for high temperature processes such as electroless nickel plating or meloniting. To determine if a coating process can be used on C64, please contact QuesTek.

    b. Consideration should be given to the time and temperature of any coating processes so that the parts are not over tempered. Contact QuesTek if there are any questions.

    c. C64 can be put through a nitrdiing process after carburizing, but nitriding is discouraged and should be attempted with caution. Previous attempts at nitriding C64 have yielded too brittle of a surface condition, leading to sub-surface spalling initiation. If nitriding is a process of interest, then a thorough investigation is strongly recommended prior to producing the final test articles or prototype parts.

    d. It is anticipated that C64 can be coated using black oxide, a conversion coating that produces a unique finish on the metal. It is recommended to conduct the black oxide coating at a temperature of 285°F (141°C), even though this process is possible at room temperature. Certain benefits of the higher temperature process are lost when conducted at room temperature. For further questions or more specific details on black oxide coating, please contact QuesTek.

    e. PVD processes applied below the tempering temperature of 925°F (496°C) should be acceptable; however if the process temperature approaches the 925°F (496°C) tempering temperature, then consideration should be given to including the PVD processing time as part of the overall 8-hour tempering cycle. PVD coating processes should not substantially exceed the 925°F (496°C) tempering temperature.

f. It is anticipated that cadmium plating or phosphate conversion coatings can be performed successfully on C64. While process specifications are not yet defined, it is recommended to perform a subsequent hydrogen bakeout process in accordance with AMS 2759/9 (using parameters outlined for AF1410).

15. Packaging:

a. Parts should be protected from corrosion prior to shipment using a light oil spray or other approved method.

b. The parts should be prepared for shipment in accordance with commercial practice and in compliance with applicable rules and regulations pertaining to the handling, packaging, and transportation of the product to ensure carrier acceptance and safe delivery.

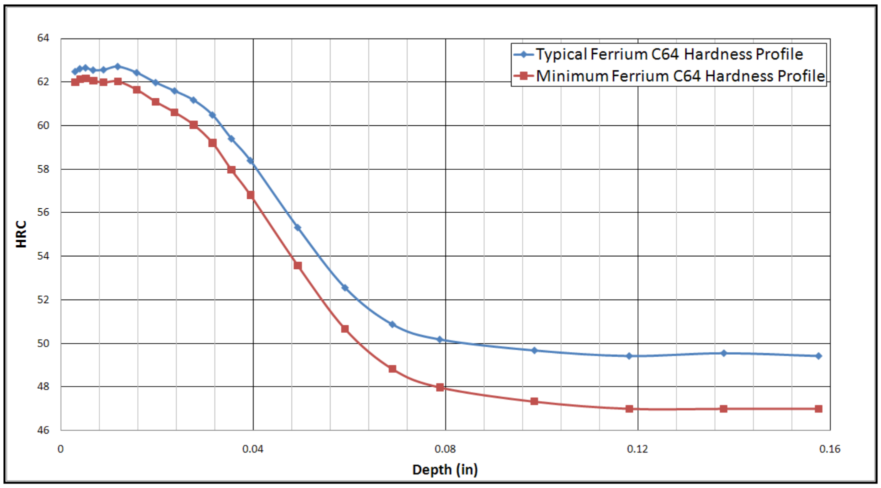

Figure 1: Typical and minimum hardness profiles for C64 for the demonstration carburization cycle discussed in Section 7.f.iii.

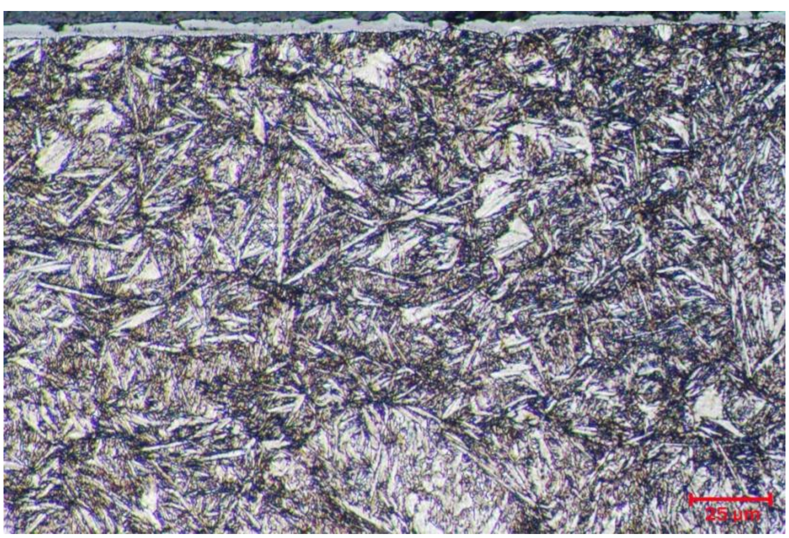

Figure 2: Typical microstructure of C64 at 400x magnification After vacuum carburization, quench, sub-zero and tempering in air as described in this specification. Sample was etched with a 2% Nital solution.

MECHANICAL PROPERTY DATA

TYPICAL CORE PROPERTIES

Test Temperature		Tensile Strength		Yield Strength		Elongation	R. of A.	Fracture Toughness	
°F	°C	ksi	MPa	ksi	MPa	(% in 1")	(%)	ksi√in	MPa√m
Room Temperature		229	1579	199	1372	18	75	85	94

Table 1: Typical mechanical properties of C64.