Processing Information

Manufacturing and Thermal Processing of *Ferrium*® M54™

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1. Purpose:
   a. This document provides introductory information regarding manufacturing and thermal processing of components made from QuesTek-designed *Ferrium* M54 steel. This information is intended to assist purchasers, fabricators and users in their application engineering of M54 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 K91973
   b. S-Basis Design Minimums are approved for use in MMPDS; A- & B-Basis Design Allowables are expected in 2013
   c. SAE AMS 6516
   d. SAE AMS 2759/3 for thermal processing
   e. SAE AMS 2759/9 for hydrogen bake out parameters
   f. SAE AMS 2759/11 for stress relieve parameters

3. Summary:

M54 is a new ultra high-strength, high-toughness, secondary-hardening martensitic steel that is produced by vacuum induction melting followed by vacuum arc remelting (*i.e.,* VIM/VAR) that exhibits very good resistance to stress corrosion cracking and grinding burn damage.

When specifically compared to AMS 6532 (*AerMet*® 100), key processing differences include:

   a. Machining operations will differ and in general M54 will be easier to machine than AMS 6532; preliminary information is provided herein.
   b. Forging process parameters will differ such that M54 has lower flow stress at a given temperature, and M54 can be forged at higher temperatures than AMS 6532. This may allow for increased throughput (reduced number of blows or reheats) and extended tooling life (reduced stresses on the dies). Forging dies previously used for AMS 6532 parts may typically be used for M54.
   c. M54 is potentially more quench rate sensitive than AMS 6532 as it relates to Charpy impact toughness (CVN), so particular attention should be paid to quench rates for M54 if CVN is important. Tensile and fracture toughness (K_{IC}) properties will have similar quench rate sensitivity as AMS 6532.
   d. M54 is less sensitive to variations in tempering time and temperature than AMS 6532, which may permit less stringent process control requirements in this regard.

When specifically compared to 4340, 300M (BS S155) or Maraging 250, key processing differences include:

   e. Machining operations will differ, and in general M54 will be more difficult to machine than 4340, 300M or Maraging 250 in the mill-annealed condition; preliminary information is provided herein. Many users rough machine M54 in the mill-annealed condition (~40-44 HRC) to remove the majority of material when M54 is softer, then heat treat the parts (to ~54 HRC), and then perform final machining.
   f. Forging process parameters will differ, and a higher forging temperature will typically be required when forging M54 in an existing die previously used for forging 4340, 300M or Maraging 250.
   g. The high solution treatment temperature of M54 at 1940°F (1060°C) can increase the propensity of parts to sag during thermal processing, so it is important to adequately support parts (especially long horizontal sections) or hang long parts vertically as feasible during thermal processing.

h. Operations such as shaft straightening (if required) should preferably be done after the sub zero treatment but prior to the temper. M54 achieves full mechanical strength after tempering, and thus trying to straighten parts after tempering will be more difficult.

i. It will typically be far more difficult to impose grinding burn damage on M54 parts, due to its higher tempering temperature (960°F or 516°C).

4. Forging

a. Please contact QuesTek to discuss your specific M54 forging application, especially when forging from ingot stock input. M54 components have been successfully forged using the general guidelines outlined below.

b. On a preliminary basis, the suggested procedure for forging M54 parts from billet and bar stock follows (if higher forging temperatures are preferred, then use the procedure in part "c" below):

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

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

iii. Normalize forgings by heating to 1965°F (1074°C) +/- 25°F (14°C), holding for 60 minutes, - 0 minutes, + 60 minutes, and then air cool to room temperature.

iv. Subcritical anneal forgings by heating to 1470°F (799°C) +/- 25°F (14°C), holding for 60 minutes, - 0 minutes, + 60 minutes, and then air cool to room temperature.

v. Anneal forgings by heating to 1205°F (652°C) +/- 50°F (28°C) for no less than 8 hours, and then air cool to room temperature.

vi. The hardness should be less than 429 HB.

c. On a preliminary basis, the suggested procedure for forging M54 parts from billet and bar stock at higher hot-fire temperatures, or from ingot stock that has been fully homogenized, is:

vii. Hot fire at 2300 to 2350°F (1260 to 1288°C), and forge with a minimum of 4:1 forging reduction ratio. The majority of the 4:1 forging ratio should be completed in the final pass in order to meet the grain size requirements.

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

ix. Normalize forgings by heating to 1965°F (1074°C) +/- 25°F (14°C), holding for a 60 minutes, - 0 minutes, + 60 minutes, and then air cool to room temperature.

x. Subcritical anneal forgings by heating to 1470°F (799°C) +/- 25°F (14°C), holding for 60 minutes, - 0 minutes, + 60 minutes, and then air cool to room temperature.

xi. Anneal forgings by heating to 1205°F (652°C) +/- 50°F (28°C) for no less than 8 hours, and then air cool to room temperature.

xii. The hardness should be less than 429 HB.

xiii. Confirm that mechanical properties (UTS, YS, elongation, RA, and 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 AMS 6532 may typically be re-used with M54. AMS 6532 utilizes a lower-temperature-stability grain-pinning dispersion particle than does M54 and is typically finish-forged no hotter than 1800°F (982°C), whereas M54 can be forged up to temperatures of 2050°F (1121°C) without concern for minimum forging reduction as it uses a higher temperature stability grain pinning dispersion particle. For this reason it may be possible to reduce (or eliminate) the number of blows or reheats required when forging M54 vs. AMS 6532.

f. Forging dies used for 4340, 300M or Maraging 250 can probably be re-used with M54, although it will probably be necessary to forge M54 at higher temperatures than 4340, 300M or Maraging 250. It is recommended to perform a trial forging after reviewing the available flow stress data. Contact QuesTek for more information.

5. Cold Working / Cold Forming

    a. There have been no prior attempts to cold-forge M54. The very high ductility and toughness of M54 suggests it should be capable of cold working. A simple, short trial run to make a few parts using M54 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. Machining

    a. M54 bar stock is typically shipped from the producer/mill in a normalized-and-annealed condition, with typical hardness of ~40-44 HRC, and with decarburization and surface defects removed.

    b. In general M54 will be more difficult to machine than the 4340 or 300M, but easier to machine than AMS 6532. A machining study is available (pending authorization to release) that details rough machining procedures for M54 in the mill-annealed condition (~40 HRC). The machining procedures covered are interrupted turning (square to round cross section), continuous turning (outer diameter), face milling, axial hole drilling (inner diameter), continuous turning (inner diameter), hole drilling and tapping, and external thread turning. The study includes insert types, coolant types, machine specifications, spindle speeds, feed rates, machining time, surface finish, and depth of cuts. Machining suggestions are also included in the study for fully heat-treated M54 (~54 HRC). The study also includes a comparison between machining procedures of M54 and AMS 6532. To obtain a copy of this machining study, please contact QuesTek.

7. Welding

    a. We are not aware of any significant investigations of welding or brazing of M54 yet. On a preliminary basis, we suggest:

        i. Making welding sticks from M54 material, then TIG weld, and then temper that part at 392°F (200°C) for 1 hour and then assess results.

        ii. Practices typically used for AMS 6532, 4340, etc. should be applicable to M54

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

    b. Please contact QuesTek for more information.

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

    a. Dimensional changes should be taken into account as the preferred manufacturing and machining paths for components are being determined. The heat treat growth/permanent expansion of M54 is isotropic and has been observed to be ~0.0010”-0.0015” per inch.

    b. 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 solution treatment, quench, sub-zero treatment and temper, 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 solution treatment, sub-zero treatment quench and temper, 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.

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

        i. If excessive distortion exists after the solution treatment, 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; a 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 not convenient to measure distortion and straighten a component before tempering, then users can investigate hot-straightening parts with greater force after tempering. It may be possible to hot-straighten M54 parts of modest size after tempering. The temperature used should be determined by amount of force required to straighten the part and should be maintained below 700°F (371°C) to avoid over-tempering or decarburization. A small oxide layer may form at this temperature. Allow the component to air cool to room temperature after hot straightening. A stress relieve process should then be completed to relieve any residual stress induced during the hot straightening process.

9. Heat Treatment

a. General Information

i. M54 can be solution treated and tempered in either vacuum, air or other media as described below, and selection of the process can depend on factors such as: industry application; ability and desire to final-machine a component after tempering; size or weight of component; achievable quench rates; etc.

ii. As with other martensitic alloys, it is recommended that fillets and rounded corners be used as feasible to limit any sharp corners or edges of components made from M54, since sharp edges or corners can serve as crack initiation points if a severe quench is used.

b. Selection of Vendor:

i. A number of heat treat vendors have experience processing M54. Where possible/feasible, QuesTek recommends that these vendors be selected for the production of initial prototype parts since they often have well-developed processes. Contact QuesTek for more information about these vendors.

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. Part Loading:

i. Parts should be loaded with no contact or stacking.

ii. It is recommended that shafts and long-sectioned parts be hung vertically, as possible, or be adequately supported. The higher solution heat treatment temperature of M54 vs. 4340 (for example) can increase the propensity of M54 parts to sag during this high temperature processing step.

iii. It is recommended that an extra carrier piece be added with a thermocouple to mimic the largest cross-section thickness and serve as the working thermocouple. If desired, one or more additional carrier pieces can be included to validate the hardness and microstructure produced during thermal processing.

e. Solution Treatment:

i. Recommended Practices for Selected Solution Treatment Method

1. Solution Treating in Vacuum is generally preferred for aerospace components, and when the parts will not be final-machined on all contact points or load-bearing/fatigue-limiting surfaces. To achieve optimum results, evacuate the chamber to at least 10^{-4} torr. Using 10^{-3} torr or other less severe vacuum levels may also yield acceptable results, but can result in a small amount of surface decarburization (e.g. ~0.001 - 0.002”).

2. Solution Treating in Air can be an acceptable alternate method when all contact points or load-bearing/fatigue-limiting surfaces will be final-machined to sufficiently remove the oxide/decarburization layer of ~0.060” that will likely occur, or for example when the size or weight of the component precludes solution treatment in vacuum.

3. Solution Treating in Endothermic Gas can be an acceptable alternative method when all contact points or load-bearing/fatigue-limiting surfaces will be final-machined to sufficiently remove the decarburization layer that will likely occur, or for example when the size or weight of the component precludes solution treatment in vacuum. To prepare the endo gas, a suggested starting point is to achieve a carbon potential of 0.1485 wt% C with reference to graphite or 0.4030 wt% C with reference to pure Fe; this can result in a decarburization layer of ~0.030” thickness. These parameters have not

been fully optimized and users are encouraged to perform tests before solution treating M54 in endo gas.

ii. Heat parts from room temperature to 1940ºF (1060ºC) in the following recommended manner, achieving uniform temperatures as feasible:

1. Pre-heat the part, preferably at 600ºF (315ºC). This preheat step is highly recommended in order to minimize the coarsening of non-desirable carbides that will be more difficult to put back into solution during subsequent solution treatment steps. In no case should the pre-heat temperature be greater than 932°F (500°C).

2. Heat from 600ºF (315ºC) to 1940ºF (1060ºC) as fast as possible in order to minimize carbide precipitation/growth, but decrease the ramp rate as the temperature nears 1940ºF (1060ºC) so as to not significantly exceed 1940ºF (1060ºC). If solution treating in air and using a separate pre-heat furnace, then first stabilize the main/gantry furnace at 1940°F (1060°C) +/- 27°F (15°C) before loading the parts from the pre-heat furnace directly into the main/gantry furnace.

One example heat-up cycle is:

1. Heat from room temperature to 600ºF (315ºC) at a rate of 800ºF/hr (444ºC/hr), and allow the part to fully equilibrate at 600ºF (315ºC).

2. Heat from 600ºF to 1900ºF (315ºC to 1038ºC) at a rate of 1800ºF/hr (1000ºC/hr).

3. Heat from 1900ºF to 1940ºF (1038ºC to 1060ºC) at a rate of 1000ºF/hr (556ºC/hr).

iii. Once the thickest section of the component has equilibrated at 1940ºF (1060ºC) +/- 27ºF (15ºC), hold for 60 minutes, -0 minutes, +30 minutes.

f. Quenching

i. Quench directly from 1940ºF (1060ºC) so that the temperature drops below 150°F (66ºC) within 1 hour using either:

1. Inert gas (Argon, Nitrogen, Helium, etc.) with a minimum pressure of 2 bar, such as when the solution treatment step is performed in vacuum; or

2. Oil or liquid quenchant, such as when the solution treatment step is performed in air or endo gas, or when the vacuum chamber has an integrated liquid quench tank. The temperature of the oil or liquid quenchant should not exceed 120°F (49°C). Use caution when quenching with water, especially large parts with large variations in section thickness.

ii. NOTE: It is important to quench M54 fast enough in order to maximize the resulting UTS, YS, K_{IC} and in particular CVN. Necessary quenching rates will differ based on part geometry and section thickness. A higher quench rate will typically result in higher mechanical properties, but also more distortion. Using higher quenching pressures such as 6 bar Nitrogen or 10 bar Helium gas quench, or an oil/liquid quench, may be desirable especially for thick-sectioned parts. Please contact QuesTek for questions regarding specific quenching procedures based on your part geometry.

g. Sub Zero Treatment (Refrigerate):

i. Within 8 hours of quenching, cool parts to -100°F (-73°C) or below and hold for a minimum of 60 minutes, followed by air warming to room temperature.

ii. 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 refrigerating.

iii. If an extended period of time is going to elapse between cryogenic treatment and tempering (e.g. 1 day or longer), it is recommended to complete a snap temper at 392ºF (200ºC) for 60 minutes to prevent cracking.

h. Tempering:

NOTE: The tempering directions listed below should be closely adhered to. While M54 is a high hardenability steel that achieves near full martensitic transformation even at slow quench rates from the solution treatment temperature, there is still some retained austenite present (even after cryogenic treatment). Therefore, rapidly cooling from the tempering temperature may result in presence of fresh martensite that may result in higher strength, lower toughness, and more distortion.

i. Recommended Practices for Selected Tempering Method

1. Tempering in <u>Vacuum</u> is generally preferred for all aerospace components, and when the parts will not be final-machined on all contact points or load-bearing/fatigue-limiting surfaces. To achieve optimum results, first evacuate the chamber to at least 10^{-4} torr; 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. Then backfill the chamber with an inert gas such as argon or nitrogen to 700 microns +/- 100 microns (in order to help accelerate the heat-up and promote temperature uniformity within the furnace).

2. Tempering in <u>Air</u> or in <u>Salt Bath</u> can be an acceptable method when up to ~0.01” of material will be removed from all contact points or load-bearing/fatigue-limiting surfaces during final machining, or if the part can be plated or covered, or if for example the size or weight of the component precludes tempering in vacuum. In addition, some color tinting of exposed metal will probably occur. One option to prevent or minimize oxidation, decarburization and color tinting during an air or salt bath temper is to apply copper-plating or stop-off paint to these surfaces (this should be applied after the solution treatment step since these materials will likely breakdown at the solution treatment temperature). To our knowledge no M54 parts have yet been tempered in a salt bath, but this is expected to be an acceptable method.

ii. Temper at 960ºF (516°C) +/- 10°F (7°C) holding for 10 hours +/- 2 hours.

iii. Cool to room temperature. DO NOT QUENCH.

1. If tempering in vacuum, then cool the part by slowly circulating gas within the chamber (at rate equivalent to air cool).

2. If tempering in air or salt bath, then cool the part in air.

iv. If the part was tempered in air or salt bath, or solution treated in air or endo gas, then machine away as desired any oxide/decarburization layer that occurred. In addition, a sandblast and oil spray procedure is recommended prior to further processing or packaging.

i. <u>Inspection of Heat Treatment:</u>

Confirm that the carrier piece achieves these minimum property requirements:

i. Hardness not lower than 53 HRC, as per SAE AMS 6516.

ii. Acceptable microstructure (typical micrograph shown in Figure 1).

iii. If test coupons are provided, mechanical testing should meet the minimum properties provided in SAE AMS 6516. Typical properties shown in Table 1.

iv. Any oxide or decarburization layer formed from solution treating in air or endothermic gas may sometimes be left “as is” if that portion of the component is not a contact point or load-bearing/fatigue-limiting surface.

v. Any oxide or decarburization layer formed from tempering in air or salt bath may sometimes be left “as is” if that portion of the component is not a contact point or load-bearing/fatigue-limiting surface.

j. <u>Carburizing and Nitriding Processes</u>

i. Carburization procedures have been successfully performed on M54, but they have not been optimized. A surface hardness of 57 HRC with a very clean microstructure has been demonstrated and a surface hardness of 60 HRC has been demonstrated but some primary carbides were formed. In both cases, the core mechanical properties remain unchanged from the typical properties. For more information, please contact QuesTek.

ii. QuesTek does not have any experience with nitriding M54 to date; computer simulations imply that it should be possible, although some nitrides may also be formed. Efforts to nitride M54 will be experimental.

10. <u>Final Machining and Warm Working</u>

a. The machining study mentioned in Section 6 also includes final machining procedures for M54 in the fully hardened state (~54 HRC): continuous turning (inner diameter), hole drilling and tapping, external thread turning, and grinding (outer diameter). The study include insert types, coolant types, machine specifications, spindle speeds, feed rates, machining time, surface finish, and depth of cuts. The study also includes a comparison between machining procedures of M54 and AMS 6532. To obtain a copy of this machining study, please contact QuesTek.

11. Peening and Other Final Cold Working Processes

a. Recommended shot peening parameters for M54 are currently being developed. Existing shot peening parameters established for 300M or AMS 6532 may be appropriate to apply to M54 parts and are listed below. Preliminary recommendations are: cast steel shot size of 230; 0.006-0.010 Almen Intensity; and 200% coverage. Contact QuesTek for more information.

b. Laser shot peening has also been performed on M54 with success and shown to increase the compressive residual stress depth compared to conventional shot peening leading to enhance fatigue properties.

12. Etching and Other Inspection

a. Etching processes successfully used on AMS 6532 can also be used on M54. For specific questions on a particular etching process, please contact QuesTek. The following details have been observed successfully using the listed corresponding etchants:

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

ii. Other microstructural detail observations (inclusions, grinding burns, martinistic substructures, primary carbides, lath packets, retained austenite and carbides, etc.) are optimized with the use of 2% Nital etch. Inspection of grinding burns with 2% Nital etch may require an extended soak time.

iii. Macrostructural detail observations (macroinclusions, segregations, etc.) are optimized with the use of 50% HCl in H_2O solution, etched at 160°F for 30 minutes.

b. Barkhausen noise inspection techniques can probably used to inspect grinding burn damage, but no BNI standards have yet been established for M54.

13. Coatings and Additional Final Processes

a. It is expected that M54 components can be coated or plated in similar manner to 300M, 4340, 4330V, AMS 6532 or other high strength steels, but consideration should be given to the time and temperature of any coating process so that the M54 parts are not over-tempered. Some examples are provided below. Contact QuesTek for more information.

b. M54 has been successfully treated with the following surface protection schemes with good adhesion:

i. Zinc-Nickel electroplating + phosphating + prime + paint

ii. Cadmium electroplating + chromate conversion + prime + paint

c. Surface protection options that have not been tested on M54 but are expected to yield satisfactory results include:

i. Coating: aluminum; chromium; nickel; prime and paint; HVOF WC-Co; silver; nickel-cadmium

ii. Phosphating coatings: Zn, Mn, and other phosphating coatings Plating: silver; nickel-cadmium

d. Hydrogen embrittlement relief bake-out processes should conform to AMS 2759/9. M54 is to be included in revision E; until it issues, it is recommended to use information prescribed for "AerMet 100" in revision D.

14. 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.

QPI-M54

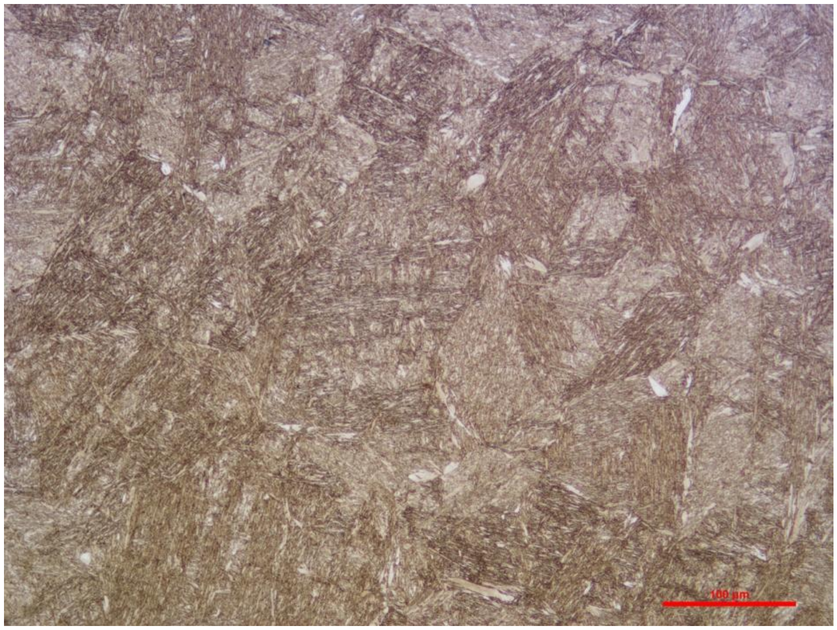

Figure 1: Typical microstructure of M54 at 200x magnification
After solution treatment, quench, sub zero treatment, and tempering in air as described in this document. Sample was etched with a 2% Nital solution.

YS (ksi)	UTS (ksi)	El (%)	Ra (%)	Hardness (HRC)	CVN (ft-lb) at RT	K_{Ic} (ksi√in)
250	293	15	61	54	24	115

Table 1: Typical mechanical properties of M54.