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Major Advances in Ferritic Nitrocarburizing (FNC)

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Introduction:

Fully automated technologies to perform ferritic nitrocarburizing or ferritic nitro-carburizing (FNC) have been utilized by Badger Metal Tech since June 2001. It was then that the equipment was first introduced to the general public. In early March 2003, Badger Metal Tech, Inc. received confirmation from the U.S. Patent and Trademark Office that **TherMaL*ife**® (FNC) is a registered process (regs. #2660875 & #2660870) for use on tool steels. **MetaLLife**® is also registered (regs. #1389443 & #2011719). The modern FNC equipment used guarantees repeatability by using a fully computer controlled and automated system. The equipment is the only one of its kind in the United States. With its nine bed stations, other types of heat treatment can also be performed. In this article, the FNC capabilities of a system which is fully automated and includes the wash, preheat, diffusion, and quench cycle will be discussed.

In this article, FNC's history, types of nitriding, the basics, controlling parameters, the operation of automated equipment, benefits received, and validation will be discussed. Since it is being used extensively in the field, the combining of (**MetaLLife**® (compressive stress texturing) and **TherMaL* ife**® (FNC) will be evaluated. By proper application, these processes provide compressive thermal crack resistance as well as protection to reduce soldering in Hot work tool steels used to cast aluminum or aluminum based alloy materials.

In the March/April 2000 issue of Die Casting Engineer magazine, a four page article was published that discussed the importance of substrate preparation. FNC is one of the pretreatment processes recommended if a subsequent coating is to be applied. This has since been validated by NADCA's Die Material Committee Coating taskforce. In addition adhesion testing was performed independently by Balzers' lab.
http://www.badgermetal.com/images/coatings/coating-adhesion-test.htm

There is new technology that enhances the future of FNC and discusses developments that are evolving in Australia. This technology uses special diffusible gases to produce a CrCN, CrC , or CrN matrix at the die's surface. Although the chemical make up is similar to PVD coatings

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being used today, they are diffused rather than deposited as done now. The result is a strong substrate with excellent adherence characteristics. You no longer have a coating but rather a diffused surface of Chromium Carbonitride (CrCN) Chromium Nitride (CrN) or Chromium Carbide (CrC). Proprietary gases, mixtures, and bed parameters are use to accomplish this.

Historic Timeline:

http://www.badgermetal.com/nitriding/nitriding-history.htm
FNC which goes by the generic name of ferritic nitro carburizing, ferritic nitro-carburizing, ferritic nitrocarburizing, and sometimes just nitrocarburizing is not new and has been around for over 100 years. Four basic properties must be present…a nitrogen source, a carbon source, heat, and some type of ferrous material.

Dr. Adolph Fry in the early 1900's found that nitrogen and iron had an affinity to one another, much the same way that aluminum and iron contact under heat can cause soldering to occur in die casting dies. From his work, in 1906 he developed the iron-nitrogen equilibrium diagram. This diagram is still valid today. If heat is applied to both iron and a nitrogen gas, the nitrogen will diffuse in the surface of the steel and along with this create a structural change in the surface of the steel affecting the hardness. This increase, it was noted, went from 282 Brinell hardness to 470 Brinell in steel containing .39% carbon and 2.88 % chromium. From this began the development of extremely high surface hardness steels called "Nitralloy" steels. These steels provided high resistance to decomposition along with being stable to temperatures up to 1800 degrees F. Dr. Fry also investigated the effect of adding other alloys such as vanadium, tungsten, manganese, molybdenum, and titanium and discovered that all of these elements would also produce stable high nitrogen content nitrides.

At the same time in New Jersey, **Adolph Machlet** while working for American Gas Company in Elizabeth, was also studying absorption of nitrogen into iron under heat conditions. He applied for his patents somewhat earlier than Fry. He consequently received his patent long before Fry on June 24, 1913. Sad to say no commercial benefits were recognized by US industries at the time, however, Dr. Jeffries in Essen Germany saw otherwise and pushed for America to further develop it.

In 1927 at an SME convention in Chicago a good friend of Fry, **Pierre Aubert** presented both the research and practical applications for it that were being used in Europe. These included, railway steel, machine tools, along with other applications in both the auto and aviation industries. Here are some of the benefits explained at the time:

- High surface hardness with practically no distortion
- Core material properties did not change
- Higher wear resistance than those achieved at the time with other surface treatments
- Tempering did not negate the hardness advantages
- No shelf life aging since parts were free of internal stress

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- Corrosion resistance

These benefits still hold true today, however, the techniques for creating the diffusion have advanced and improved. Work continued and in 1928, **McQuaid and Ketchum** both metallurgists at Timken - Detroit Axle Co. presented yet another paper using the work of Fry and Machlet as their pivot point along with some of their own investigative work into practical applications and costs.

Next in 1929, **Robert Sergeson** from Central Alloy Steel Corp. in Canton, Ohio did work that was published with regard to the effect of varying the aluminum content of the nitralloy along with the effect of nickel.

It was not until V.O. Homberg and J.P. Walsted at MIT that any work was done on the effect of varied temperature on the physical properties of the nitriding steels, equipment preheat-treatment, and decarburization effects on nitrided steel. In their work, the initial comments were made regarding the phenomena of "white layer" and its effects on component performance.

Dr. Carl F. Floe an Associate Professor at MIT continued the study of the "white layer" (epsilon) effect and supported discussions of various methods to change the composition and reducing or changing this thin hard layer. His work today is known as the "The Floe Process".

All of this early pioneering work in the field of nitriding ranging from process control, evaluation of metallurgical results, alloy steel developments, and most others still are gospel in today's processing environments.

This early work led the way for the 1930 Plasma Ion Nitriding or "Glow Discharge Technique" as it is also known. The jump start for this technology was the desire to be able to shorten cycle times, reduce distortion and improve upon the metallurgical problems and properties associated with nitriding.

Continuing development, refinement, and modern day capabilities have progressed nitriding from the early liquid, gas, and plasma to a FNC fluidized bed method. Fluidized solids exist in nature, such as quicksand. Fluidization is also not a new technology with an American patent dating back to 1879 which describes the roasting of minerals under fluidized bed conditions. The patent draws attention to the temperature uniformity provided by such methods of heating material. **Now in the 21st Century comes fully automated fluidized bed FNC technology.**

Types:

http://www.badgermetal.com/nitriding/carburizing-types.htm#top

Fluidized bed FNC is a thermo-chemical process where nitrogen, carbon, and to a very small degree oxygen atoms diffuse into the surface of the ferrous substrate forming a compound layer and subsurface diffusion layer. Objectively the diffusion is accomplished in a relatively short period of time and at sub-critical temperatures. The resulting diffusion zone is subjacent to a surface "white layer" (compound or epsilon layer), as it is sometimes called. The **primary**

objectives of FNC is to improve wear properties, corrosion resistance, and improve fatigue characteristics of the metal.

About 55 years ago commercial methods of ferritic nitrocarburizing in molten salts came into existence on a commercial basis. The first process in France was called "Sulfiniz" and used cyanide and cyanates salts plus a small amount of sulfur bearing compounds. In 1959 a modified version patent from Germany was filed for an aeration oxygen source which was called "Tufftride". The 1970's EPA regulations impacted the cyanide based materials and new salt compositions were developed that eliminated this deadly, caustic, and carcinogenic material. The new non-cyanide salt was called Melonite along with a French process called "Sur-sulf". These two salt bath processes are still being used commercially today. Through the years many different recipes have been developed but generically they are still diffusing nitrogen and carbon.

Criteria:

http://www.badgermetal.com/nitriding/fluid-bed-criteria.htm

Fluidization of the bed

Fluidization consists of making a bed of dry finely-divided particles, usually aluminum oxide, behave like a liquid. This is accomplished by moving gases through the medium's particles which separates them microscopically as the gases are fed through the bed. The majority of fluidized beds used for heat treatment are of the aggregative or bubbling bed type. The smooth or bubbly properties of the solids and fluids will determine the fluidization quality. On the other hand, the size of the bubbles and heterogeneity in the bed influences the rate of solid mixing. These factors include bed geometry, gas flow rate, type of gas distributor, and internal vessel features such as baffles, screens, and heat exchangers. It can easily be seen that the success of fluidization, carbonitriding, and quality of treatment is influenced by many needed control factors. ThermaLife® has these controls.

Gas velocities v/s temperatures

If you plunge your hand into a fluidized bed medium (preferably one that is not heated), the sensation is the same as placing your hand in a bucket of water. When an aluminum oxide medium is fluidized in a bed, and a light object is introduced in the bed, it will float the same as in a liquid. The required flow of gas is inversely proportional to the temperature of the bed and decreases rapidly with temperature. **Graph #1** shows the effect of temperature on gas flow and heat transfer rate with 1mm diameter particles that have an apparent assumed density within the desired range. The higher the temperature the lower the needed gas flow to maximize fluidization. You must also keep in mind that heat transfer rate coefficient is affected by gas velocity. This will be explained later. Also one must not forget that fluidization needs to be maintained at a temperature that is below the critical temperature of the steel (last heat treat temper temperature). This prevents the possibility of compromising the initial heat treat tool hardness. **Chart #1** shows the importance of various controls for repeatability"

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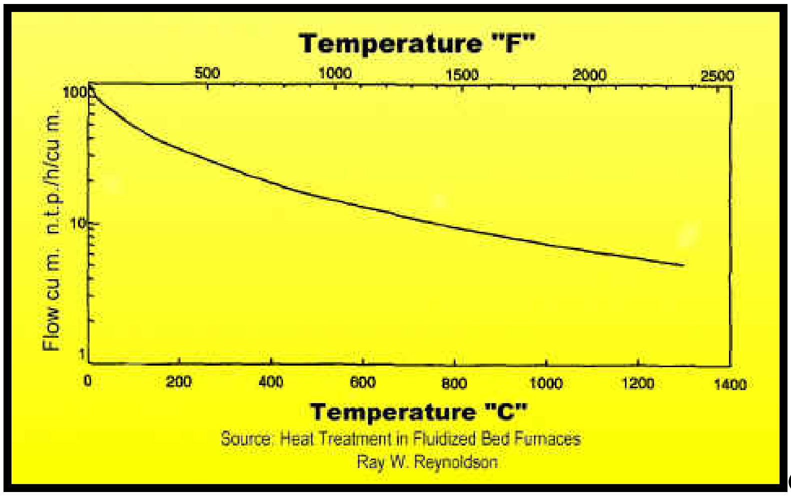

Graph #1

As temperature increases	↑	**required gas flow decreases**	↓
Too Low of temperature-clumping	↓	**reduced heat transfer coefficient**	↓
Too High of temperature -cavitation	↑	**reduced heat transfer coefficient**	↓
Too High of temperature	↑	**steel hardness may be affected**	↓

(Donna, you will may have to recreate this spreadsheet if it is not a high enough resolution)

Chart #1

Heat Transfer Factors:

http://www.badgermetal.com/nitriding/fluid-bed-criteria.htm#heat%20transfer

Three main factors affect the heat transfer rate in a fluidized bed. It is not enough to just maintain temperature. All of these items are also critical to optimization of the process and the development of the correct recipe for each specific tool steel.

Cleanliness of the tool steel - First and foremost is the cleanliness of the part or tool steel. Usually a mild to aggressive alkaline bath at elevated temperature is recommended to remove both water and oil base materials on the surface and any other areas that will contact the bed media. If these trace elements are not entirely removed, the nitrocarburizing may be compromised even though some scrubbing action is accomplished during fluidization. Without proper cleaning there is a risk of contaminating the bed media. For these reasons it is important to ask and know how the tools are cleaned prior to processing. **It is also imperative that USED tooling have the tool shop or die caster remove all of the solder by polishing or using a NaOH solution.** Something as simple as this can cause immediate failure of the nitrocarburizing as well as inconsistent unexplained results.

Particle Diameter - This parameter has the greatest influence on heat transfer. Tests have shown that the medium should be as small as possible. In practice the optimum size is 100 micro mm (3940 micro inches).

Bed Material Density - It has been determined that the governing physical property of the bed material is the density. The apparent optimum value is around 1280-1600 kg/cu m or about 80-100lb/cu ft. Denser materials tend to give a lower heat transfer coefficient and require more gas flow for fluidization. Sometimes this condition can be exacerbated to an electrostatic effect if too low of density material is used. Importantly other properties such as thermal conductivity and specific heat do not enter into the equation and are relatively unimportant.

Fluidization Velocity of Gas/Gases:

http://www.badgermetal.com/nitriding/fluid-bed-criteria.htm#critical

Fluidization Velocity of Gas/Gases - To obtain the maximum heat transfer, it is essential that an optimum flow rate be used. This generally is between two and three times the minimum fluidization velocity. Too high of velocity leads to particle entrapment, a higher consumption of fluidizing gas/gases, and poor heat transfer. Too low of velocity gives poor heat transfer and lack of uniformity of processing. A fully automated and computer controlled FNC process, such as used by **TherMaL*ife**®, regulates this and all the other critical requirements to assure a repeatable highly controlled ferritic nitrocarburizing process.

Conventional methods involving manual operations and older beds, attempt to control these criteria by using a large number of screws in the bottom of the bed. The passage of the gas between the threads of these screws control how much gas is introduced into the system. These screws loosen and/or tighten over time which drastically affect the heat transfer rate between the threads. Because of the labor intensive aspect of the maintenance of these screws, which require that the bed be totally emptied, adjustments are often neglected or bypassed. **It is important to investigate this especially if one sees unexplained variations in processing occurring. Our equipment, a multi-station bed does not use screws.** Our automated equipment uses a series of adjustable ceramic screens and eclipse valves with flow meters and sensors which by computer programming provide the exact control of the gas velocities to provide optimization of temperature vs velocity.

Graph #2 shows the relationship of velocity to heat-transfer coefficient. This data clearly shows that there is a definite optimum gas/gases velocity that maximizes the essential heat transfer rate.

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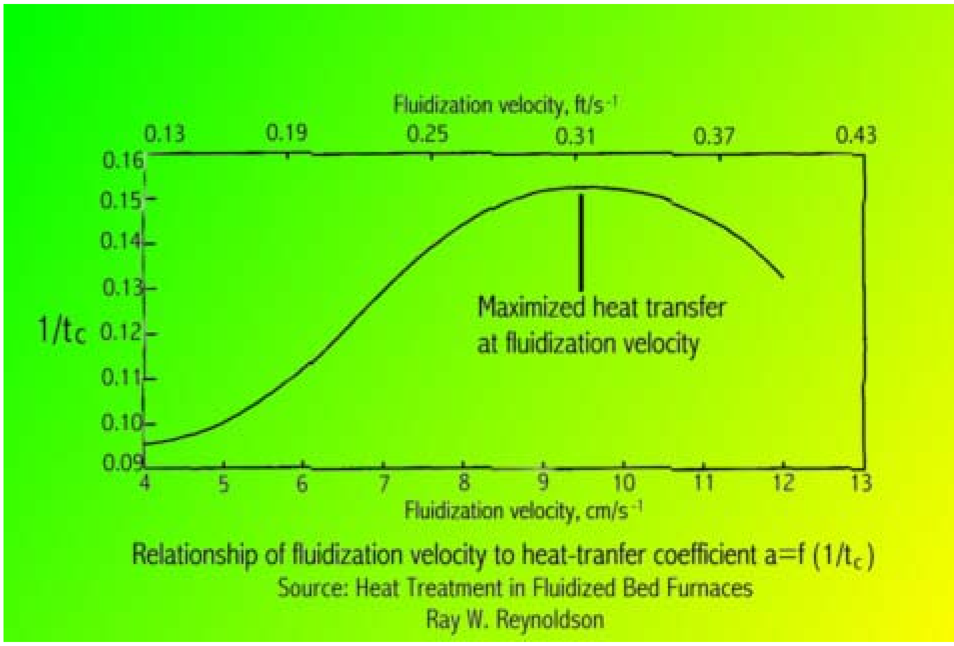

Graph #2

Gas Composition and percentage - When all of the above have been taken into consideration, the correct recipe of the gases used and percent amounts of each for diffusion of the elements must be present.. There must be a nitrogen source, such as ammonia (NH_3) and a carbon source such as CO or Natural Gas (CH_4) for FNC diffusion to occur..

Automating the FNC diffusion:

http://www.badgermetal.com/fluidized-equip/tl-equip-info.htm

The automated system, which is about 100ft x 20ft x 10ft, consists of 9 beds each with a diameter of 700mm (28”) and a draw of 1200mm (48”) **(see photos 1-4).** Maximum load is 800 kg (1760lbs) which is determined by the transport system. **(see photos 5-8)**

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Photo 1
looking down the line

Photo #2
bed stations

Photo 3
diffusion stations

Photo 4
fluidized bed retainer

Photo 5
loading station

Photo 6
top of 6 position loading carousel

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Photo 7
carousel latching mechanism

Photo 8
six station load carousel transporter.

The automation process begins with selection of the proper recipe from a database of choices. The ingredients of the recipe include diffusion/clean/quench times, temperature, gas composition and percentages, flow rate, as well as all the other variables previously discussed. A CRT monitor on the front of the control panel allows for real time viewing of the complete process **(see photos 9-14).** The CRT has nine screen options so that all or any single station can be entirely monitored and data collected. There are GUI readouts of temperature per bed which is held in computer and thermocouple controlled close tolerances. Other operating conditions of the bed including status, gas flow, velocity, media fill, transport action, and time at operating temperature are automatically monitored with corrections made as necessary to assure optimum parameter compliance.

Photo 9
CRT and station monitors

Photo 10
CRT with real time viewing.

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Photo 11
bed monitoring panel

Photo 12
transport and clamping monitor

Photo 13
gas velocity and percentage controls

Photo 14
overall view of all 9 stations status

The load is set which is the only manual operation **(see photos 16-20).** Once this is completed and the recipe has been selected, the loads program is initialized. The next time that the load is manually handled is when it is off loaded from the carousel after quenching. A variety of basket configurations are selected depending on the size and weight of the tooling being processed.

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Photo 16
open basket for larger inserts and tooling

Photo 17
multi-level loaded basket

photo 18
mesh construction for smaller tools

Photo 19
load with latch pin on carousel

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Photo 20
transport track to 6 position carousel

Sealing of any of the heated beds not being used is accomplished by covering them with special steel covers. The covers have locators on them so the transport can remove them. These are either stored in the carousel assembly or with a load on the carousel. The transporter first takes the cover off of the heated bed to be used and replaces it with the load which also has the same cover. Once in place, a computer links to the thermocouples at a number of strategic locations in the bed. The desired temperature of the bed is tracked and when equilibrium has been reached between the load and bed media, the diffusion process starts. All of this is accomplished by computer controls monitored through one of the 9 screen views at the control panel's CRT. Once the diffusion process is completed, the load is moved by the transporter to the quenching bed where other gases are used, and the load is then removed. **(See photos 21-25)**

Photo 21
bed lid for both empty bed and load

Photo 22
transporter delivering load

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Photo 23
placing load in bed for diffusion


Photo 24
removing load from bed


Photo 25
none FNC high temperature operation

Microstructure benefits:

http://www.badgermetal.com/nitriding/table-diffusion-treatments.htm

Since the load is totally immersed, there are no "line of site" limitations during the diffusion process. The well controlled sub critical temperatures also do not allow any compromise of the final heat treatment tempering. Knowing the final temper temperature assures both carbon and nitrogen atoms, obtained from the chemical properties of the gases, are diffused into the tool steels surface. Because of the uniformity of heat transfer, the process is accomplished in a short period of time. During the process a diffused zone and an outermost micro thin hard beneficial layer, referred to as the "white layer", "epsilon", or "compound layer", is formed.

The depth of both of these areas is relational to the amount of Cr, Ni, Al, and other alloys that make up the steel, the temperature of the bed, the time in the bed, and the types/amounts/velocities of the gases used. **(See photo 26-27)**

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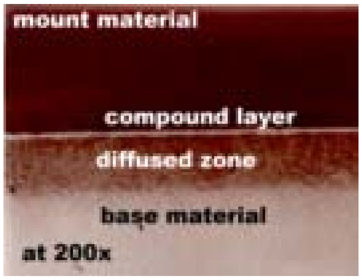


Photo 26
short time and lower temperature

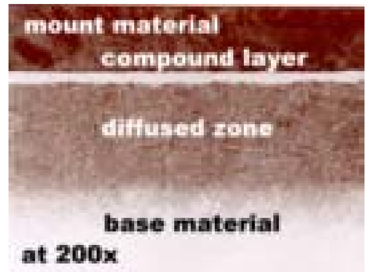
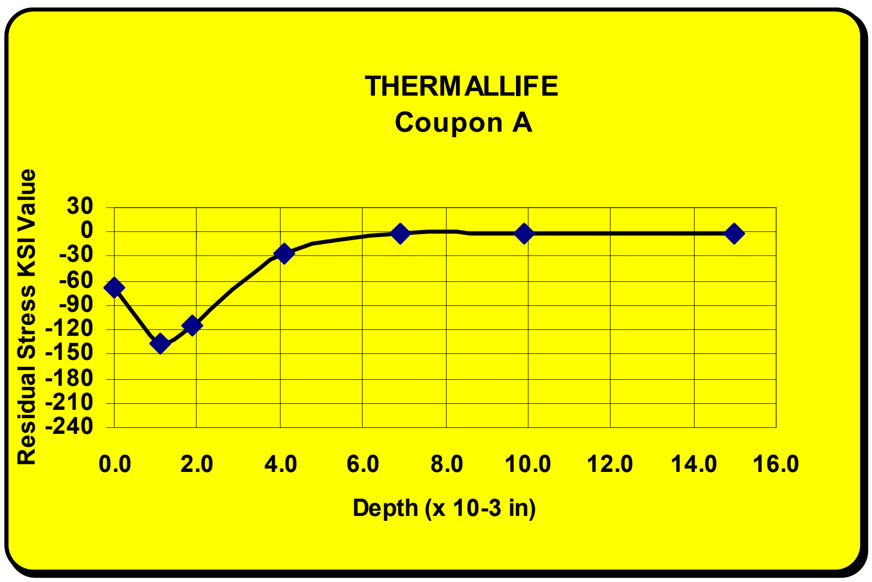


Photo 27
same steel higher parameters

Some minor compression is introduced in the diffusion zone that is not very deep but high in magnitude just below the surface. **(See graph #3)**



graph #3
high compressive depth in diffusion zone but shallow

The compound layer hardness is usually in the range of 1000 Knoop (10g). This and the diffusion layer act as a barrier to prevent the soldering of the aluminum content in the cast metal from interacting with the die's surface. Micro-hardness readings show how rapidly the hardness drops off below the diffusion zone. **(See photo 28 & graph 4 and photo 29 & graph 5)**

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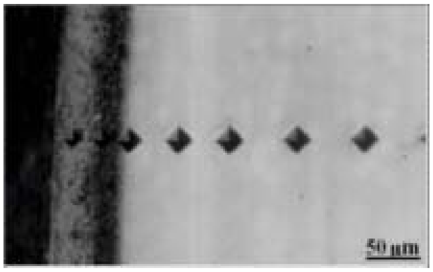


photo 28
shallow hardness diffusion zone

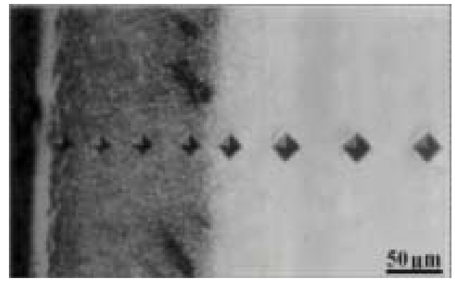
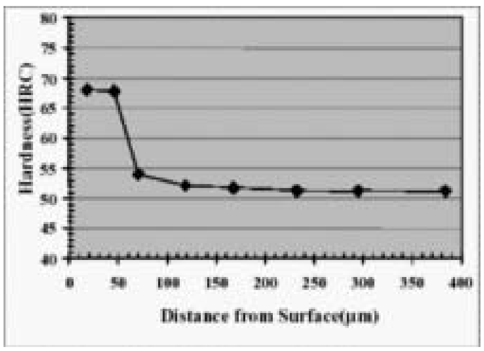
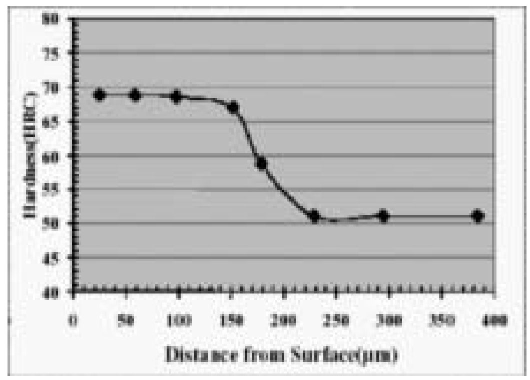


photo 29
deeper hardness diffusion zone



graph 4



graph 5

Validation by a recent study of the soldering characteristic of aluminum was performed by Case Western Reserve University and can be viewed on the web at:

http://www.badgermetal.com/ml-dc/solder-test/solder.htm.

The study showed that if the diffusion zone is too deep, the embrittlement of the surface could lead to premature cracking or chipping of the steel's surface below the compound layer. Thinner diffusion zones and compound zones seemed to provide the best protection against both soldering and thermal fatigue cracking. Photos comparisons can be viewed at:

http://www.badgermetal.com/ml-dc/solder-test/solder-summary.htm
http://www.badgermetal.com/ml-dc/solder-test/solder-thick-vs-thin.htm

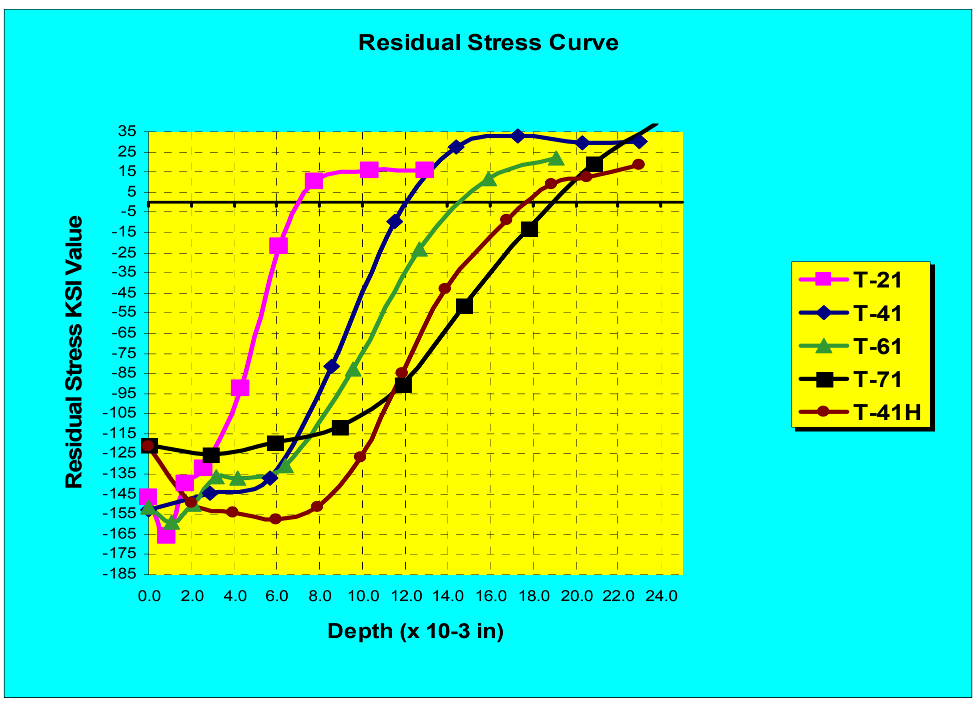
It should be noted that the newer H-13 steels with a chromium matrix will FNC more quickly at lower temperatures and times. It is therefore extremely important to query steel companies producing such steels. FNC processes then need to be adjusted according to the specifications received.

The final color of the FNC can be controlled by gases used during processing and at quench. A deep blue/black color results when tooling is dynamically steam blued. Other colors are possible with other gases. The color adds an oxidized surface only for appearance purposes which does not affect the FNC process. Without any quench color added, the steel is a semi gloss deep gray to dull black color. If there is a doubt about the FNC white layer being properly created, special chemical salts can be used. The chemical causes a color change on the steel's surface indicating the areas where the compound layer is not present.

Combining FNC with existing technologies

Since the early part of 2002, customers have been combining FNC with compressive stress texturing and receiving the double benefit of crack and solder prevention. Accepting the metallurgical principle that a crack will not propagate through a compressive zone until the fatigue strength of the material is exceeded, one can see that the compressive stress is a benefit. Varied mechanical stress texturing creates different levels of compression at a significant depth as the following X-ray diffraction curves (all processes combined) illustrate. (See graph #6) For separate graphs of each process go to:

http://www.badgermetal.com/bmtcurves/lambda-metallife-curves.htm



graph 6
X-ray diffraction curves showing depth and amount of compression

Following this premise, coupons for X-ray diffraction were sent to an outside lab for measuring after compressive stress texturing & FNC had been applied. Four different coupon combinations were created…1ea. only FNC process, 1ea. T-41 first followed by FNC, 1ea. FNC followed by T-41H, and 1ea. FNC followed by T-41. The measurements were taken until a depth of

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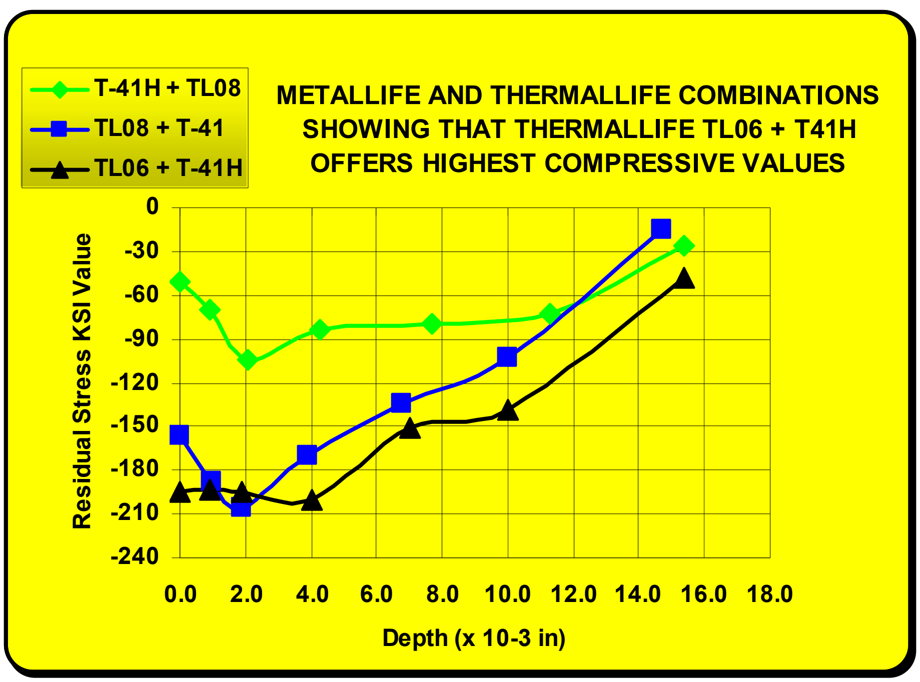
approximately .015" was reached. The goal was to answer two questions…1. What effect did the heat from FNC have on the previously applied mechanical compression? 2. How much compression would result if the FNC was done after stress texturing? In the case of the "FNC followed by T-41H, the recipe had to be adjusted to prevent spalling of the compound layer.

When the results were tabulated and graphed, it was noted that the FNC temperature soak time did not relieve all of the compression in the coupon that was first T-41H processed then FNC treated. The two coupons that were first FNC treated and then T-41 or T-41H processed exhibited a high level and depth of compression with the T-41H combination coupon **exhibiting higher compression than either the FNC or T-41H alone**. Because of these test confirmations, it is now common practice to apply both of these processes to field tooling. More information regarding these tests along with a full set of graphs of the results can be found at:

http://www.badgermetal.com/lambda-coupons.htm

Reference the summary **graph #7.** Note how, in the case of the TL-06 + T-41H, the compression value at the surface exceeds either process being separately applied. Also the compressive depth is significant to about .012" to 014" of an inch.



Graph #7
The black line (TL06 + T-41H) highest compression value (ksi) and depth

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The pathway to future diffusion methods

At present Physical Vapor Deposition (PVD) is used to develop a chromium carbide (CrC), chromium nitride (CrN), or other alloy surface coating. An Australian firm has refined a method of doing this by diffusion. The project is already past the research and development stage. The apparatus is similar to that shown in this article. The difference lies in the media and gases that diffuse metallic elements into steel surfaces at sub critical temperatures.

As a result of the project, provisional patents have been registered, and commercial equipment is available to perform a new range of low temperature surface treatments known under the generic trade name of Qab.

In principal, using specially constructed fluidized beds and a proprietary media and gas mixture, the heated part is diffused within a special bed. Special bed material, gases, and elements are used for the reactive purposes. Some elements that can be diffused include Chromium and Titanium. The net effect is a diffused treatment that mimics the characteristics of PVD CrC, CrN or other coatings while providing the needed adhesion and substrate structure. More detailed information can be found at: http://www.badgermetal.com/qab-related/qab.htm.

Conclusive summary:

Ferritic Nitro carburizing (FNC) in a fluidized bed is not new technology, however, new methods are being used today that assure better control for defined repeatability and consistency. FNC is a thermo-chemical process where nitrogen, carbon, and to a very small degree oxygen atoms diffuse into the surface of the ferrous substrate forming a subsurface diffusion zone and sometimes a white layer on the surface. The primary objective of FNC is to improve wear properties, corrosion resistance, and improve fatigue characteristics of ferrous metals. The presence of the white layer and diffusion zone assists in reducing the tendency for aluminum to solder to the tool steel surface which eventually forms an inter-metallic bond of the two materials. Because the fluidized bed allows for complete immersion of the tool steel, heating of the tool is more uniform with no "line of site" limitations as encountered with PVD coatings. Unlike CVD coatings that are done at extremely high temperatures, the sub critical temperature at which FNC is accomplished eliminates the danger of annealing or distortion of the tooling.

Gas velocities, percentages, temperature, time, and cleanliness are all interdependent on one another and determine the final outcome. Controlling these is best accomplished by using fully computer controlled and automated equipment. Various recipes can be used to develop the diffusion zone and white layer by adjusting the aforementioned criteria. The benefits of FNC with regards to reducing soldering have been validated by Case Western University.

TherMaL*ife® can be combined with **MetaLife**®. This affords both a solder barrier and high value in the compressive zone with depth. The two processes assist in protecting the tool against both the fatigue and solder failure mode. Field tests have validated the benefit of doing this procedure.

The future for FNC is bright since technology has been developed and machinery is available that mimics today's PVD coatings but accomplishes it by diffusion rather than vapor deposition in a vacuum. In principal, using specially constructed fluidized beds and a proprietary media and gas mixture, the heated part is diffused with elements of Nitrogen, Carbon, and whatever other elements that are used for the reactive process. Chromium and Titanium are some of the elements that could be used. The net effect is a diffused treatment that provides both the needed adhesion and substrate structure. More detailed information can be found at: http://www.badgermetal.com/qab-related/qab.htm.

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