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Continuous Case Hardening of PM Gears with Low Pressure Gas Quenching

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Abstract

An effort was made to find an alternative to the conventional practice of case hardening sintered parts in batch and continuous heat treatment furnaces with oil quenching. Trials were conducted for case hardening (carburizing and carbonitriding) of PM gears in a continuous sintering type of furnace. The heat treatment atmospheres used were endogas as well as N_2+H_2, in both cases with enrichment (doping) hydrocarbon & ammonia gas. A predominantly martensite case was achieved in gears made in Astalloy 85Mo. Paper describes process conditions and properties achieved in terms of surface hardness, hardness profile, surface carbon potential and tooth breaking strength. A comparison of mechanical and fatigue properties was made against conventionally heat-treated gears. The case was more clearly defined than generally seen in oil quenched parts.

Introduction

Manufacture of automotive gears by the powder metallurgy route represents a significant reduction in both the part cost as well as the carbon footprint of the manufacturing process due to the elimination of several steps by this near net shape process. Astalloy 85Mo (0.85% Molybdenum and 0.2% Carbon) from Hoganas AB is a suitable material for gears. This alloy needs to be case hardened properly to obtain good fatigue strength, surface hardness and tooth strength for its successful application as gear components.

PM components are conventionally case hardened by heating the parts in batch & continuous furnaces with endogas atmosphere operating typically at 850ºC to 930ºC followed by oil quenching. Due to interconnected porosity, it is difficult to achieve a defined case (carbon diffused surface layer depth) profile. In addition, thin sections get through carburized & therefore become brittle due to high core hardness. Later when the parts are tempered the oil within the parts burns & causes smoke & pollution unless the tempering furnace is specially designed with an after burner.

Low pressure carburizing (LPC) with high pressure gas quenching eliminates several of these problems [1] but the capital cost is high & the productivity is low and therefore this seems suitable only for parts with high value addition.

An alternative new processing method has been developed in the Fluidtherm R&D Center where PM gears are case hardened in a continuous mesh belt furnace (like a conventional sintering furnace) and then gas quenched (at atmospheric pressure) in a sinter hardening type module (Fig.1). From purely a productivity viewpoint it would take several LPC furnaces to produce quantities like 300 Kg/Hr which is easily achieved in a single continuous belt furnace of this type.

Figure 1: A typical continuous sintering furnace with gas quenching module

Process development

Two types of gears, both made from Astalloy 85Mo were obtained from two different manufacturers. Figure 2 shows a gear with a density of 6.9 g/cc (a) from Sintercom P.Ltd, India and a gear with a density of 7.1g/cc (b) from Hoganas India Ltd.

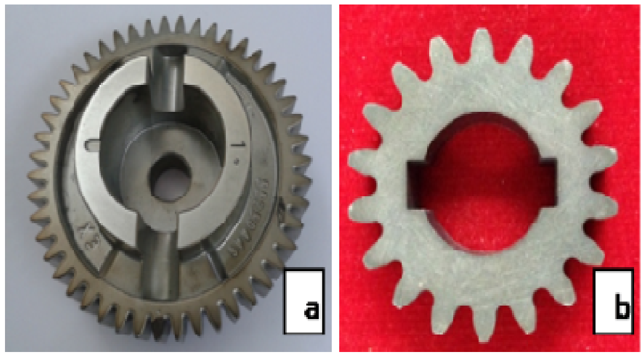


Figure 2 Astalloy 85Mo components from Sintercom (a) and Hoganas (b)

The hardenability of the case matrix was altered by using modification of the furnace atmosphere constituents, location of injection points, flow rate & flow direction. This 'improved' case hardening process was carried out in two types of furnace atmosphere systems, one a nonstoichiometric mixture of N_2+15%H_2 as carrier gas and the other in a synthetic endogas derived from independently cracked methanol and nitrogen. All trials were carried out at temperatures of 930ºC to 980℃ and the soaking time was varied between 50-90 minutes. Gas quenching was carried out at a cooling rate of 7°C/second to 8ºC/second, monitored by embedded trailing thermocouples hooked to a data logger.

The gears thus hardened and subzero treated were tested for hardness, case depth, microstructure, and tooth breaking strength after tempering (all gears) at 180ºC for 60 minutes. Carbon concentration gradient was determined by spectroscopic analysis of the case-hardened surface. Tooth breaking test was carried out as per Sintercom specifications.

Fatigue testing was carried out in a FZG-gear test rig at the Hoganas India Ltd. laboratory by subjecting the processed gears to a stress range of 1200 to 1450MPa at various cycles.

Results and discussion

Trials with improved furnace atmosphere conditions with the non-stoichiometric N_2+H_2 atmosphere plus the required enrichment gases showed good case hardening characteristics (Fig. 3).

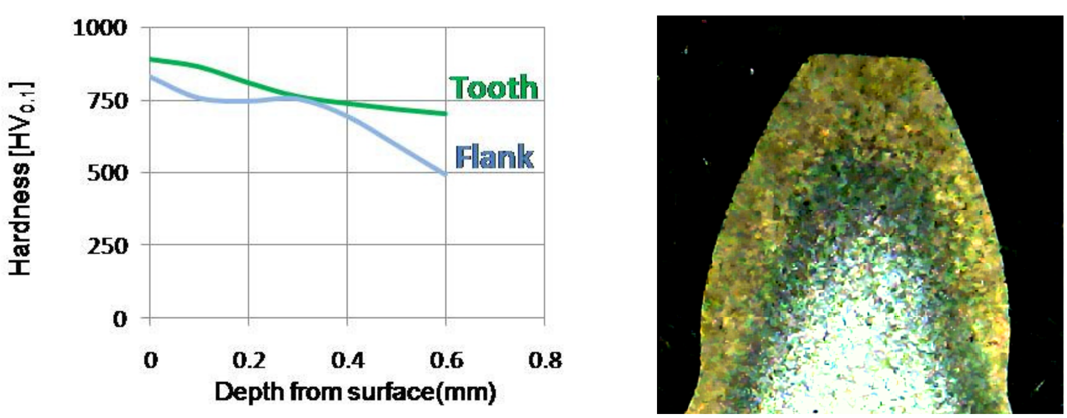


Figure 3 Hardness profile and case structure of the improved case hardening trial, surface carbon potential of 0.85-0.95%C.

The case structure in initial trials showed the presence of 10 -20% retained austenite. This was rectified by sub-zero treatment < (-) 70℃ prior to tempering at 180℃ for 60 minutes. The case structure in the modified trials shows predominantly tempered martensite with insignificant retained austenite. Core microstructure shows ferrite with fine pearlite (Fig. 4).

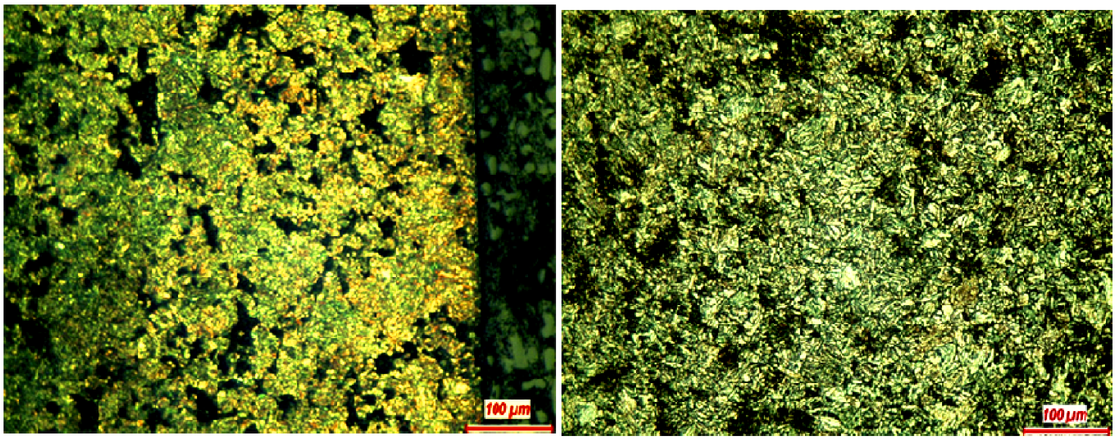


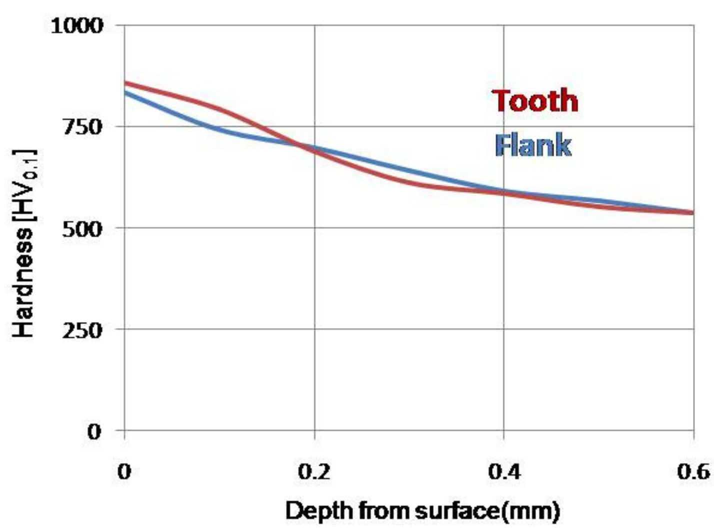
Figure 4 Tooth flank (left) and core (right) microstructure, (surface carbon potential 0.85-0.95%C)

Table 1 gives the flank hardness comparison between Sintercom specifications (from conventional case hardening practice) and as actually achieved. As can be seen the values satisfied the minimum requirements.

Hardness	Specification (Tooth Flank)	Achieved (Tooth Flank)
At the surface	800 - 850 $HV_{0.1}$	830 $HV_{0.1}$
At 0.5mm case depth	550$HV_{0.1}$	594$HV_{0.1}$
In the Core	325 $HV_{0.1}$ Max	179-229 $HV_{0.1}$

Table 1 Hardness comparison

Case hardening trials in synthetic endogas stoichiometric atmosphere also showed good case hardening characteristics (Fig. 5).



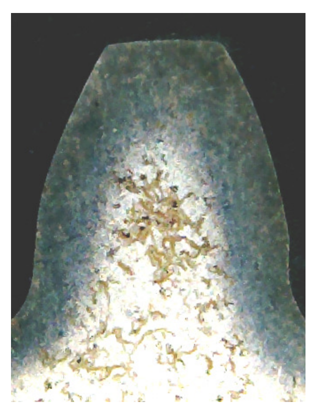


Figure 5 Hardness profile and case structure of the trial in synthetic endogas atmosphere, surface carbon potential of 0.85-0.95%C.

In this case also the presence of 10 - 20% retained austenite was seen & converted to martensite by sub-zero treatment < (-) 70℃ prior to tempering at 180℃ for 60 minutes. The case structure after tempering shows predominantly tempered martensite with insignificant retained austenite. Core microstructure shows ferrite with fine pearlite (Fig.6).

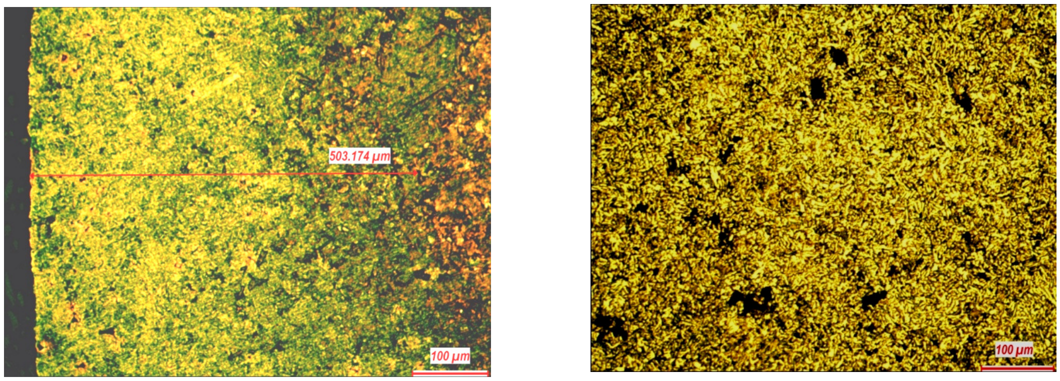


Figure 6 Case (left) and core (right) microstructure (surface carbon potential 0.85-0.95%C)

Carbon Potential profile

Carbon concentration gradients were measured on gears processed with both atmospheres (non-stoichiometric and endogas – Fig. 7)

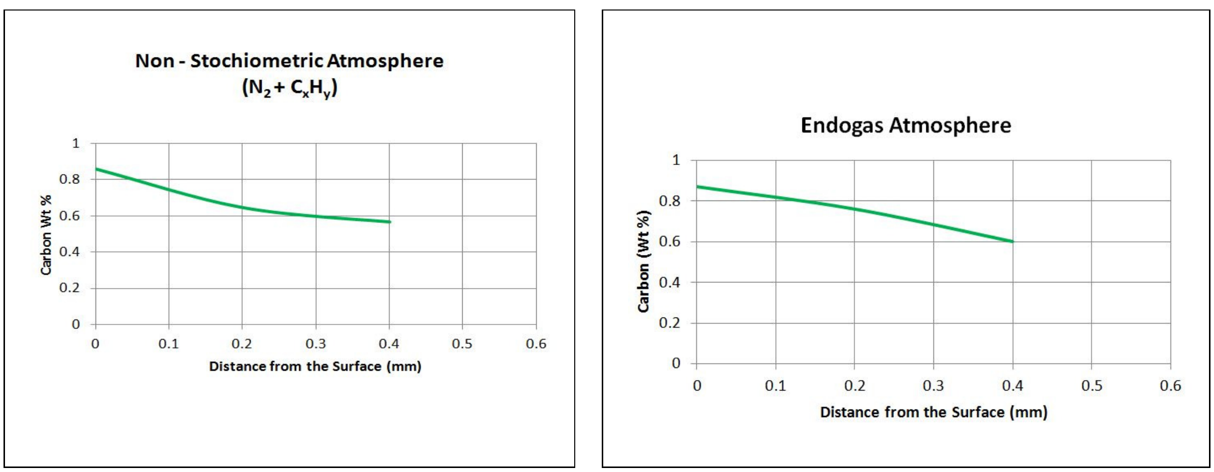


Figure 7

Fatigue test results

Gear's heat treated at Fluidtherm (Continuous Carbonitriding with Gas Quenching - C+AGQ) have similar fatigue life as compared to the gears heat treated by LPC with Oil Quenching (Hoganas) -Fig.8.

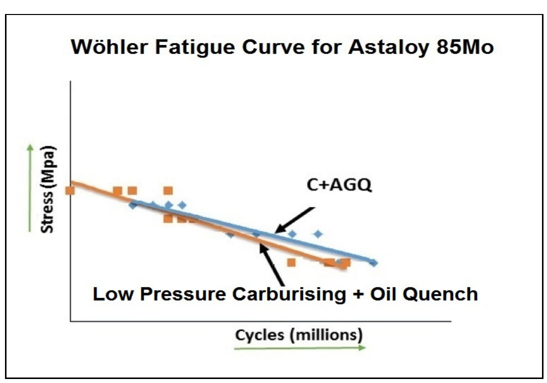


Figure 8 Wöhler curve

The tooth breaking strength was studied on the Sintercom gear cam shaft. The test fixture is shown on the left of Fig. 9 and a gear with a cleanly broken tooth is shown on the right. The tooth breaking strength was recorded as 8.3 KN. This is comparable to other gears sintered in the same lot which were conventionally case hardened & oil quenched.

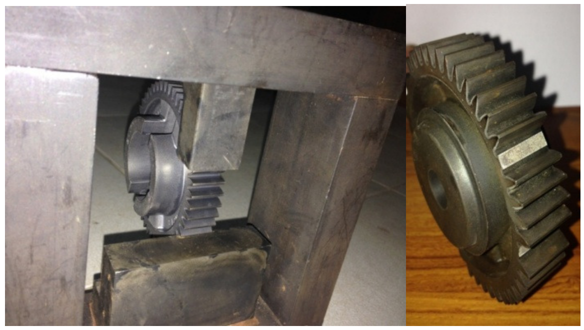


Figure 9 Tooth strength test setup (left) and specimen after test (right)

Conclusion

Continuous furnaces like conventional mesh belt sintering furnaces with gas quenching facilities similar to conventional sinter hardening modules are effective for case hardening PM gears made in Astalloy85 & other alloys with similar hardenability.

Similar level of surface hardness, microstructure and core hardness is achieved in two different furnace atmosphere systems, non-stoichiometric N_2+H2 and stoichiometric endogas, both with required enrichment gases.

C+AGQ process when compared with conventionally case hardened & oil quenched parts.

- Well defined case structure & a sharp hardness differential between case & core.
- Tooth strength is satisfactory and comparable.
- Comparable fatigue test results.

The successful use of non-stoichiometric N_2+H_2 (with enrichment gases) opens up a possibility of elimination of internal oxidation especially of high chromium PM parts.

The core hardness of processed parts was seen to be lower than oil quenched parts & this is naturally due to the lower heat extraction rate of convection at atmospheric pressure. Where & if at all required this can be increased by a higher carbon content or more alloying or by low pressure quenching at under 3 barg in a modified continuous furnace.

Reference

1. Magnus Dahlstrom, M. L. (17 September 2013). High Performance PM Components Heat Treated by Low Pressure Carburizing and Gas Quenching . *Presented at EURO PM 2013 in Gothenburg* , 1-6.

PROCESS PROTOTYPING FOR PRODUCTION SCALE SINTER (BAINITE) HARDENING OF PM PARTS BY GAS QUENCHING

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ABSTRACT: The microstructure of sintered PM parts is generally ferrite and pearlite and the pearlite content is in proportion to the carbon content. This can be changed to martensite by the hardening process done by heating the parts & rapid cooling either in oil or under pressurized & recirculated gas. PM parts can also hardened by 'sinter hardening' in a wire mesh belt sintering furnace within a module placed immediately after the furnace. The furnace atmosphere is re-circulated across heat exchangers & directed on parts as they emerge from the sintering furnace. Generally high alloy compositions (which harden easily) are required to obtain a predominantly martensitic structure as the cooling capability of recirculated gas is lower than oil or pressurized gas. Several machined or stamped (not PM) spring steel parts as well as cast ductile iron parts are hardened in a manner so a bainitic structure is formed instead of a martensitic structure (for improved fatigue properties). This is done by austenitising the parts & quenching them to 280°C to 400°C & holding them till complete transformation to bainite is achieved. This process is called Bainite Hardening or Austempering. Other researchers (1,2) have described methods of obtaining a bainitic microstructure in PM parts by suitable alloying & cooling rate control and have reported the resulting properties obtained. However, there are very few references on the results of conventional austempering performed by isothermal holding at the required temperature. It was decided to build an (open to public) process prototyping furnace to perform sintering followed by rapid cooling to the required lower temperature & iso-thermal holding between 250°C to 500°C. The purpose was to study the response of different PM alloys and part geometries. As a first trial gears made in Astalloy CRM + 0.5%C & Astaloy Mo + 0.6%C were processed and the results are reported in this paper.

Keywords:

Bainite Hardening

Gas quenching

Sinter hardening

PM Austempering

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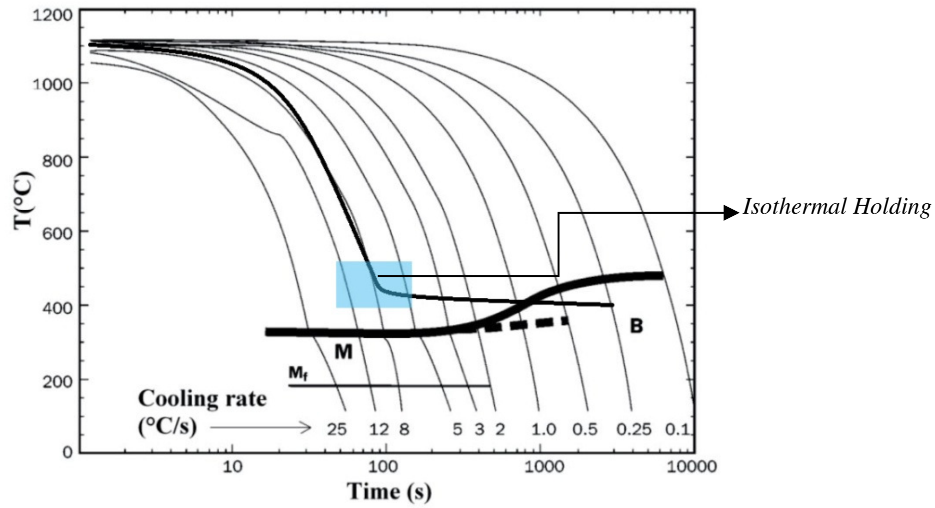
1. Experimental methods

A study of CCT diagram of CRM alloy (kindly provided by Hoganas AB) was made with a view to estimate the process parameters & implement these in the prototype furnace & study the different types of bainite obtained. Fig. 1 shows the CCT curve of CRM alloy & on this is superimposed the desired cooling curve.



M=Martensite, B=Bainite

Fig.1 CCT Diagram for Astaloy CRM

This shows that rapid cooling at a rate of 2°C/sec & higher would result in a martensite structure. A slower cooling rate of 0.5°C/sec to 2°C/sec may result in bainite but with martensite patches. In both cases the parts would need to be tempered to remove stresses caused by the formation of martensite. If full bainite transformation (upper or lower) for CRM alloy is required, the parts need to be cooled to 350°C/sec to 400°C/sec rapidly and then isothermally held for the time required for 100% bainite transformation.

The resulting bainite may be upper bainite or lower bainite depending upon the isothermal holding temperature. Tempering can be eliminated if there is no martensite formed.

2. Experimental setup:

A sintering furnace with a gas quench module was constructed for the study. The furnace concept and the required sintering & cooling profile are shown in Fig.2*)*.

Fig.2 Sinter (Bainite) Hardening furnace with Thermal Profile

The parts are transported through the sintering furnace for debinding & sintering prior to entry into a gas quench module and then a convection low temperature furnace for isothermal holding.

First trials (Fig. 3) were performed in an atmosphere of 90% N_2+ 10% H_2. The test parts were loaded on a wire mesh tray (Fig.3) and sintered at 1120°C for 25 minutes and then moved to the gas quench chamber (Fig. 3). The cooling rate was measured by a single trailing thermocouple. The quench rate was controlled by varying re-circulation fan speeds & the dwell time in the quench chamber.

The parts were cooled to 310°C & hold there for 30 minutes (Fig. 4).

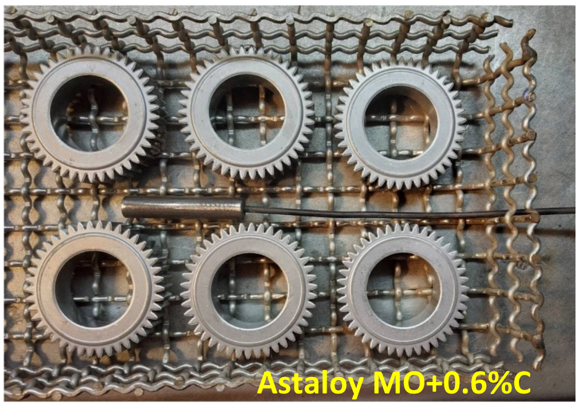


Fig.3 Part loaded on a wire mesh tray with embedded trailing thermocouple

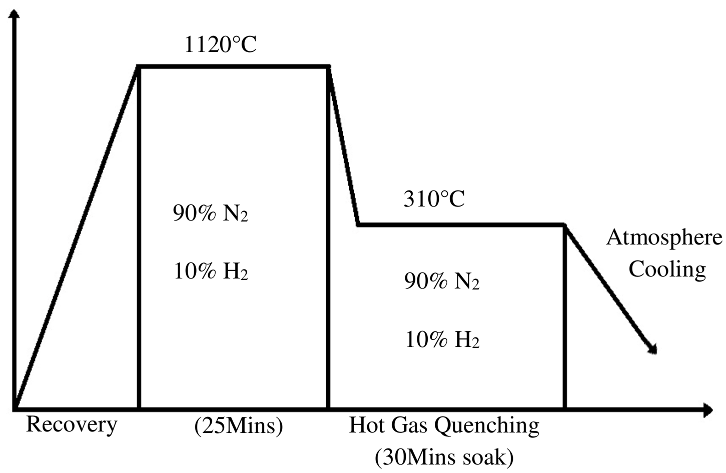


Fig.4 Trial process cycle

3. Results:

3.1 The measured temperature profile & cooling rate is superimposed on the ASTALLOY CRM CCT curve are shown in fig.5

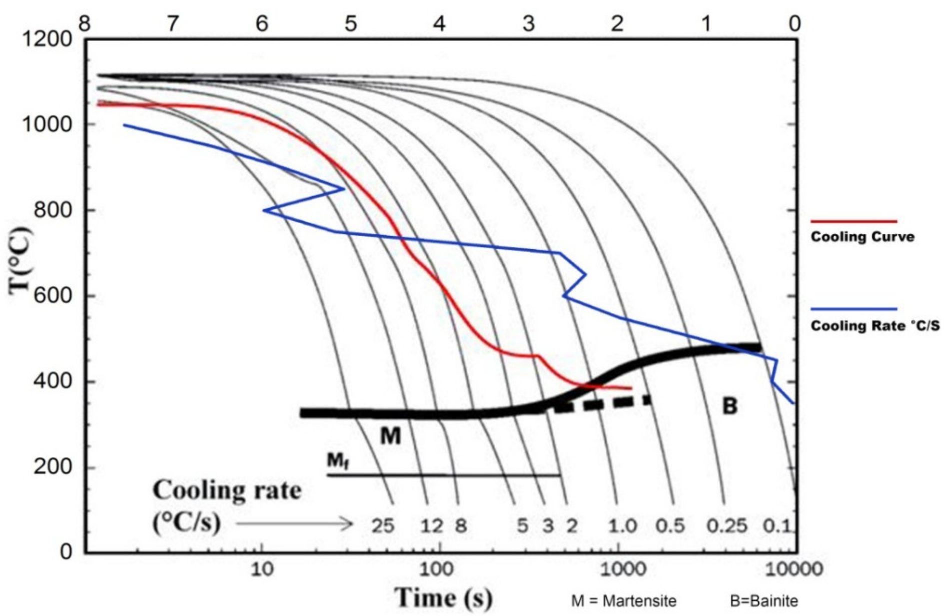


Fig.5

3.2 Microphotographs:

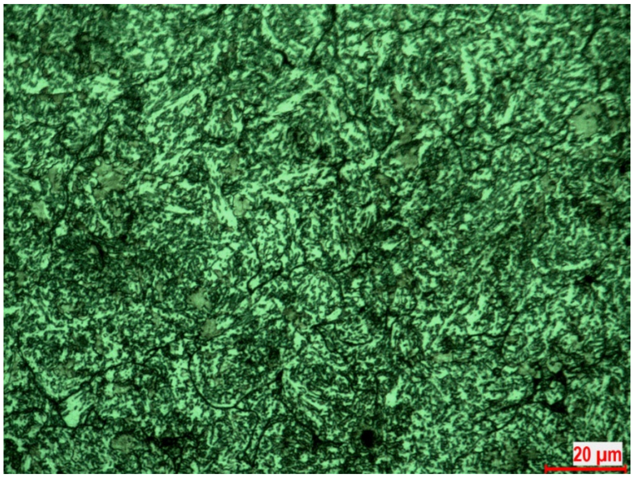


Fig.6 Astalloy CRM Microstructure shows lower bainite with some ferrite and without any trace of martensite.

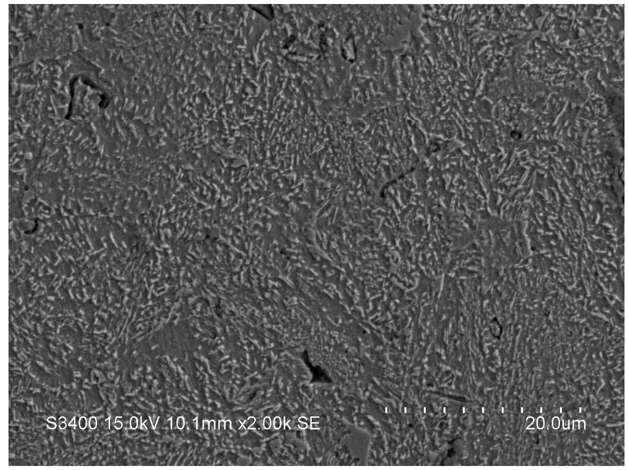


Fig.7 Astalloy CRM SEM Micrograph shows predominantly bainitic structure.

Hardness: 350 HV 0.1

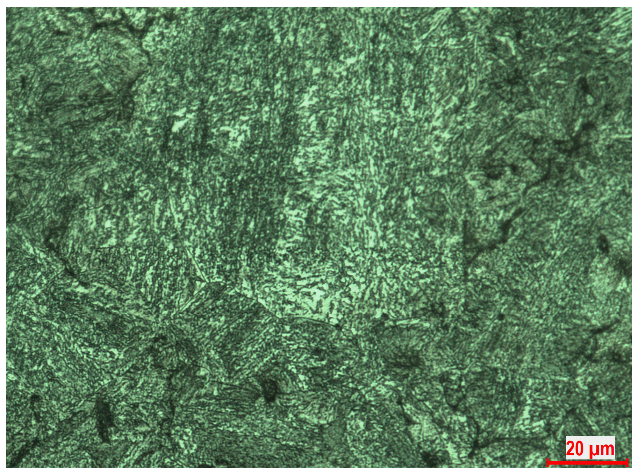


Fig.8 Astaloy Mo + 0.6C Microstructure: Predominantly upper bainite with some ferrite and without any trace of martensite.

Hardness: 320-380 HV 0.1

3.3 Hardness (Astaloy Mo+0.6C)

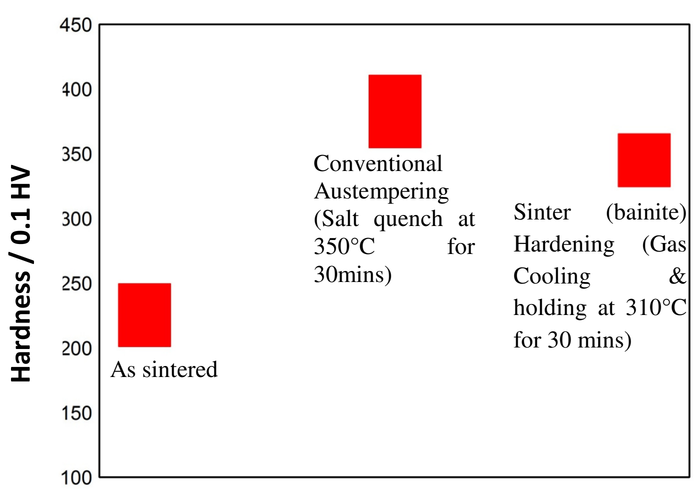


Fig.9

4. Conclusion

Preliminary trials show that predominantly bainitic structure can be achieved on ASTALLOY CRM and ASTALLOY Mo sintered parts by rapid cooling & isothermal holding at the bainite transformation temperature. The prototype furnace is being used to carry out sinter (Bainite) hardening trails for a variety of parts under different processing conditions.

References

[1] Schade C, Murphy T, Alan Lawley A, Doherty R. Microstructure and mechanical properties of a bainitic PM Steel;. Philadelphia, PA. 2015. p. 1–14.

[2] Sulowski, M.: Arch. Metall. Mater., vol. 59, 2014, no. 4, p. 1499

THE LOW GAS CONSUMPTION CONTINUOUS FURNACE: ADVANTAGES OF THIS NEW FURNACE CONCEPT FOR PM APPLICATIONS

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PMAI. PM 2021. International conference on Powder Metallurgy & Particulate Materials

Abstract
When a need was seen for a furnace with an increased cooling rate and reduced gas consumption, a (patent applied) Low Gas Consumption furnace concept was developed. The LGC furnace is a hybrid of mesh belt and pusher furnaces, utilising the best aspects of each to create a new product. The authors report on the goals behind the LGC furnace's development, and what can be achieved using this new furnace concept.

The LGC furnace: Evolution

The motivation behind the development of the LGC Furnace was to increase the flow through the sinter hardening cooling blower in mesh belt sintering furnaces to the cooling rate.
This new design concept also reduces gas consumption substantially and makes it possible to change the way several thermal processes are performed.
Mesh belt furnaces used for processes like debinding, sintering, steam treatment, annealing, normalising and brazing – are, in principle, tunnels, open at both ends and having sufficient headroom to allow a conveyor belt loaded with parts to travel into and out of the furnace (Fig. 1). Typically, the amount of gas these furnaces consume is in proportion to the cross-sections of the tunnel opening.

Fig. 1 A conveyer belt loaded with parts passes into a mesh belt furnace (Courtesy Fluidtherm)

The quantity of gas required for blanketing or for reaction with the parts' surfaces in a mesh belt furnace is only a small fraction of the amount of gas consumed; most goes towards keeping air out of the furnace tunnel. Other furnaces – such as pushers, walking beams and roller hearths – utilise atmospheric lock doors to keep air out of the furnace, thus substantially reducing gas consumption when compared with an equivalent capacity mesh belt furnace.

To address the considerable difference in gas consumption between these furnace types a decision was made to add atmospheric locks to a mesh belt furnace.

Steel, ceramic or graphite trays are required to carry the parts through the LGC furnace; these trays enter the furnace and exit sideways, passing through atmospheric locks at both the entry and exit points.
(Fig. 2 left and right, respectively).

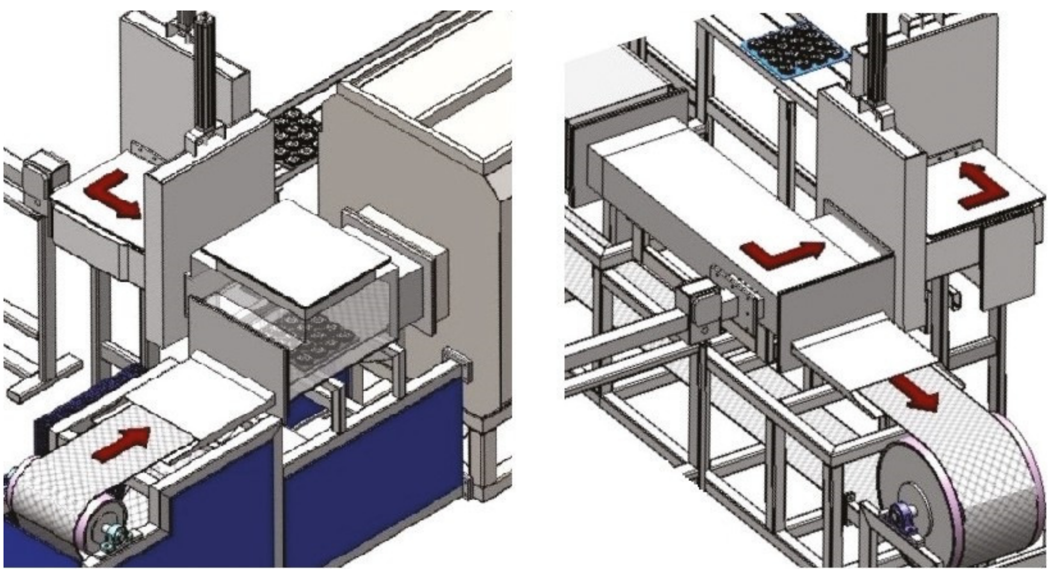


Fig. 2 Part-carrying trays enter and exit the LGC furnace sideways, passing through atmospheric locks at both the entry and exit point

With this side entrance layout, the belt is empty at the place where it enters and exits the furnace tunnel, allowing the front and rear openings to be only a few millimetres taller than the belt itself without the need for head- room (Fig. 3). This reduces the gas consumption to the low levels seen in pusher furnaces.

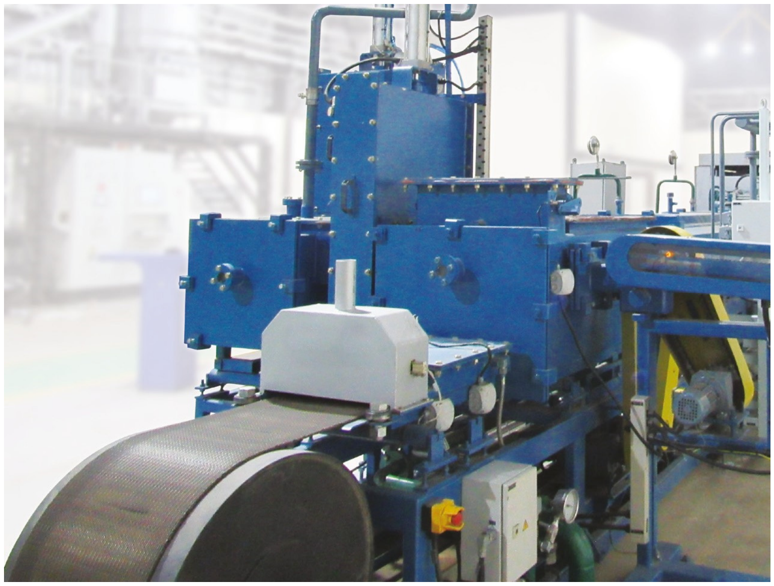


Fig. 3 Because parts enter and exit the furnace via a side entrance on the LGC, the front and rear openings through which the conveyer belt passes do not require the same headroom as in a typical mesh belt furnace

WHY NOT JUST USE A PUSHER FURNACE?

The question that might arise when considering the development of the LGC furnace is what its applications might be. In fact, LGC furnaces which are hybrid mesh belt/pusher furnace combine the advantages of both; over mesh belts in terms of gas saving and over pusher furnaces in terms of a reduction in the deadweight of the trays (typically, LGC trays weigh a third of pusher trays); the elimination of tray pile up; not having to feed empty trays to empty the tunnel of parts; the elimination of problems caused by undulations in the pusher track, in the case of older metal alloy muffle furnaces; and the possibility of using higher-capacity plants, with trays for 600 mm to 900 mm wide belts.

This new Low Gas Consumption design opens up multiple possibilities.

Reduction in Cost of Thermal Processing

Gas consumption is the second highest component of the overall cost of most thermal processes, next only to heating energy cost. Any reduction in the gas cost has a significant impact on the overall processing cost. The energy needed to heat unnecessary gas to the furnace temperature must also be considered.

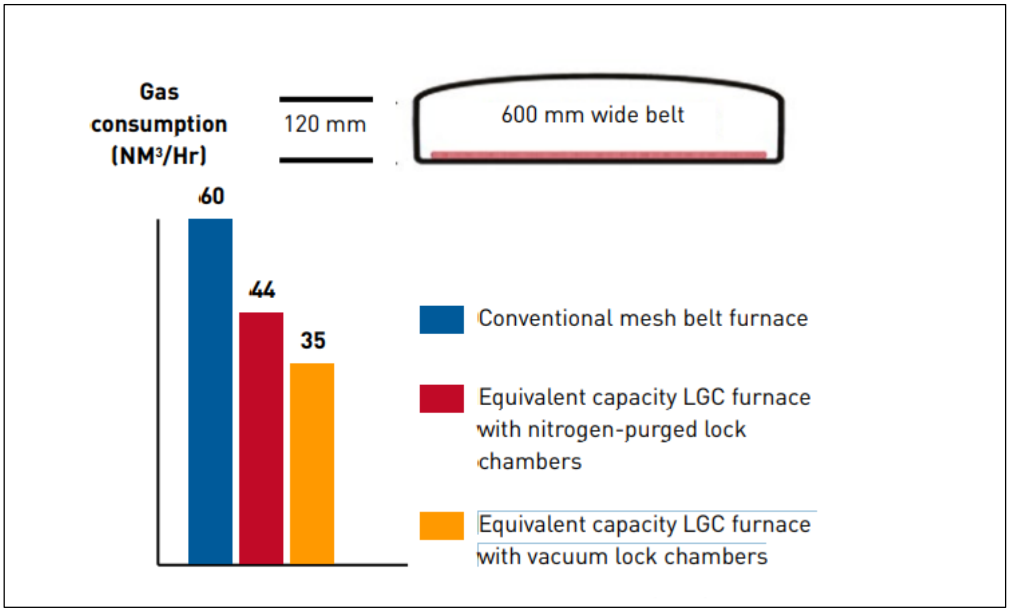


Fig. 4 Gas consumption in a conventional mesh belt furnace (blue), equivalent capacity LGC furnace with nitrogen-purged lock chambers (red) and equivalentcapacity LGC furnace with vacuum lock chambers (orange)

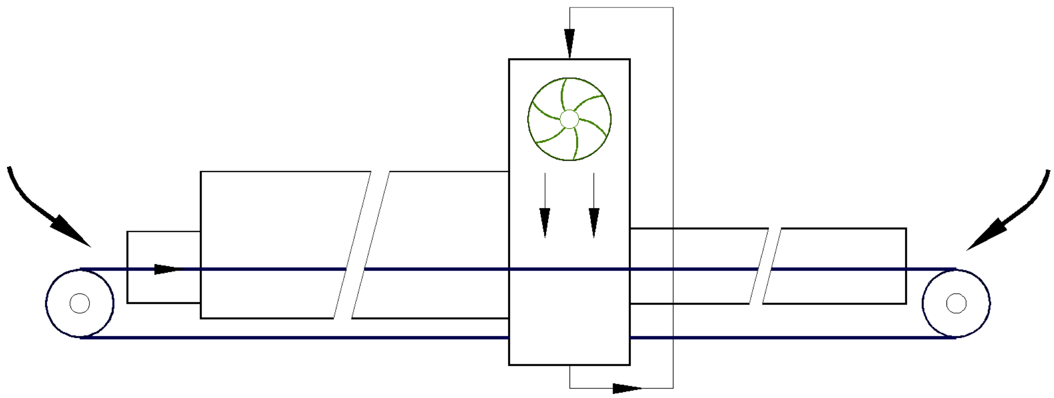
The gas consumption of an LGC furnace equipped with nitrogen- purged atmospheric lock chambers is about 27% lower than a traditional 300 kg/hour net, 600 mm wide belt furnace operating with a mixture of N_2 + 10% H_2. When the LGC furnace is equipped with vacuum atmospheric lock chambers, the gas consumption reduces by 42% (Fig. 4).

Unlike conventional mesh belt furnaces, increasing the LGC furnace size (belt width & tunnel length) does not cause a proportionate increase of gas consumption. Consequently, the cost saving due to reduced gas consumption increases with an increase in furnace capacity. The cost saving increases dramatically when the content of expensive hydrogen in the gas mixture is high for instance, when copper brazing or sintering stainless steel.

SINTER HARDENING IN MESH BELT FURNACES:

Sinter hardening occurs when the furnace gas is recirculated across a cooler and is impinged on the surface of hot sintered parts as they emerge from the sintering furnace. The performance of a sinter hardening module is considered satisfactory if heat is extracted from the parts at a rate of around 3℃/sec.

If the fan speed is increased in an effort to increase the cooling rate, air can often enter into the furnace tunnel, resulting in oxidation of the sintered parts despite blocking systems (Fig. 5).



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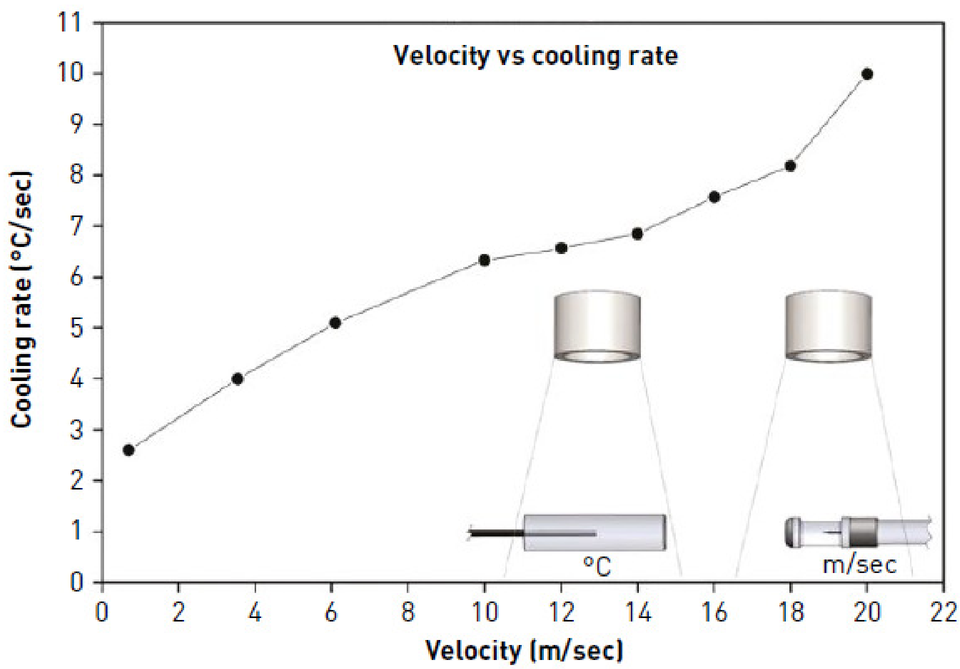

Fig. 6 Velocity vs cooling rate test. A Ø12.5 mm x 50 mm Inconel rod with a Ø2.6 mm N thermocouple inserted to the centre was heated to 1080 ℃ and subjected to a nitrogen stream from a D50 tube nozzle placed 120 mm above the rod

When the cross section of the entrance and exit is severely reduced, as in the LGC furnace concept, the chance for air to enter the tunnel is correspondingly reduced. The sinter hardening blower is then able to work at a higher speed and the untapped potential of higher velocity gas impingement (Fig. 6), which would result in a higher cooling rate, can be realised. This has the potential to reduce the cost of certain PM parts by reducing the extent of expensive hardenability enhancing elements.

Continuous Carburising Of PM Gears With Low Pressure Gas Quenching:

At the 2016 World PM congress in Hamburg, Germany, the authors presented a paper discussing the viability of carburising PM gears in a continuous mesh belt furnace, similar to a sintering furnace, followed with hardening by gas quenching at atmospheric pressure in a sinter hardening module [1]

Results were reported on the testing of PM gears made from Höganäs' Astalloy 85Mo powder, carburised and hardened at a cooling rate of 8℃/sec in a prototype furnace.

The LGC furnace, with cooling rates higher than those generally achieved in a mesh belt sinter hardening furnace, will make the continuous carburising of PM gears viable at an industrial scale. One such continuous furnace has the capacity to replace the several batch atmosphere and vacuum furnaces currently used in this application and at a lower processing cost. This was, in fact, the primary reason for the development of the LGC furnace concept.

Continuous Furnace Brazing Of Tall Parts: *Several parts that are brazed – such as small gas cylinders and tube manifolds – sit tall on the furnace belt. The consumption of hydrogen- rich gases in a continuous furnace for brazing such parts would be high, if not for the 'humpback' construction of inclined and extended front and rear tunnels, which reduce gas consumption by half. However, there are limits to the angle of inclination to prevent parts sliding down the furnace belt; this increases the plant length (Fig. 7). One of the consequences of this extra length is the added cost of replacement belts, which are sold by the metre. The installation of a long hump back furnace can also become a problem when manufacturing floor space is inadequate.*

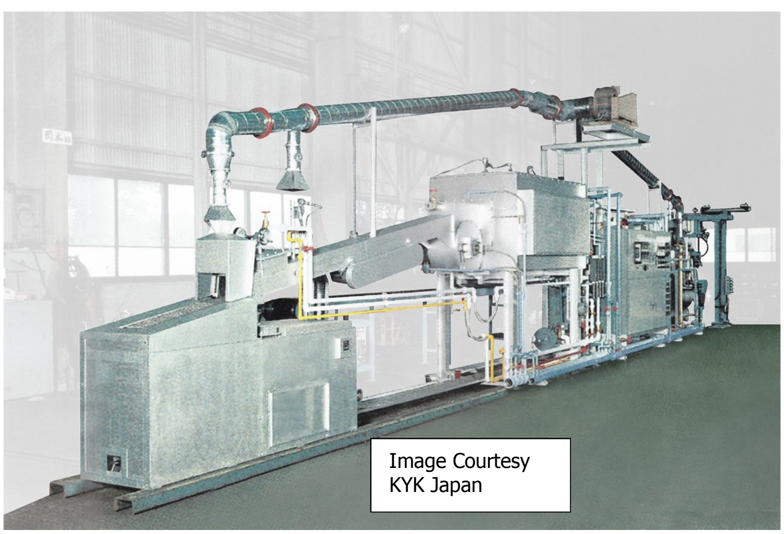

Fig. 7 The 'humpback' construction of inclined and extended front and rear tunnels in continuous brazing furnaces reduces gas consumption by half, but there are limits to the angle of inclination to prevent parts sliding down the furnace belt.

An LGC brazing furnace of the same capacity is shorter, as the inclined entrance and exit tunnels are replaced by the shorter atmospheric lock chambers, which allow increased headroom without a proportionate increase in gas consumption.

Continuous Steam Treatment Without Boilers:

Many companies, which need to maintain boilers for process steam, would be receptive to the idea of a steam treatment furnace that does not require a boiler. On top of the problems related to boiler safety and maintenance, one has to consider the cost of the energy required for the latent heat of trans-formation of water to steam.
In a conventional mesh belt steam treatment furnace with an open entrance and exit, the process steam from a well-maintained boiler is an economical option for the amount of steam the furnace consumes. As is the case with most processes conducted in these conventional furnaces, the amount of steam actually required for the process of oxidising the PM parts is a relatively small fraction of the total quantity of steam used, most of which serves the function of keeping air out of the furnace tunnel.

When the gas consumption is reduced in an LGC furnace with entry and exit atmospheric lock chambers, it becomes cost-efficient to use nitrogen gas saturated with water vapour in a heated bubbler (humidifier) in place of steam.

A purpose-designed humidifier, equipped with special tubular heaters and droplet capture, gives the same result as steam from a boiler for both shock absorber and refrigeration parts. The ability to add a ‘finishing’ step involving the direct injection of an oxygen source makes it especially useful for parts subjected to high hydraulic pressures, as well as parts that require a particularly lustrous blue-black hue.

Sintering of Aluminium Parts:

The purity of the protective nitrogen atmosphere in an aluminium sintering furnace has to be of a higher order than what is required for iron parts, considering the affinity of aluminium for oxygen [2].

In this case, the LGC furnace would have, in addition to the atmosphere lock chambers that reduce air in the tunnel, an all-metal hot zone comprising a metal alloy muffle to prevent reactions with the furnace insulation, recirculation fans for improving temperature uniformity in multi-tier work baskets, in-muffle heaters that are isolated from the parts for convective heat transfer without hot spots, and atmosphere isolation that keeps binder fumes away from the sintering chamber [3].

Soft magnetic alloy processing:

An LGC furnace built with features suitable for aluminium sintering, as well as for steam treatment, also becomes suitable for the stress relaxation of soft magnetic alloy parts where required, with atmosphere isolated sections for binder removal, inert gas and high-dew point (steam) atmosphere (Fig. 8).

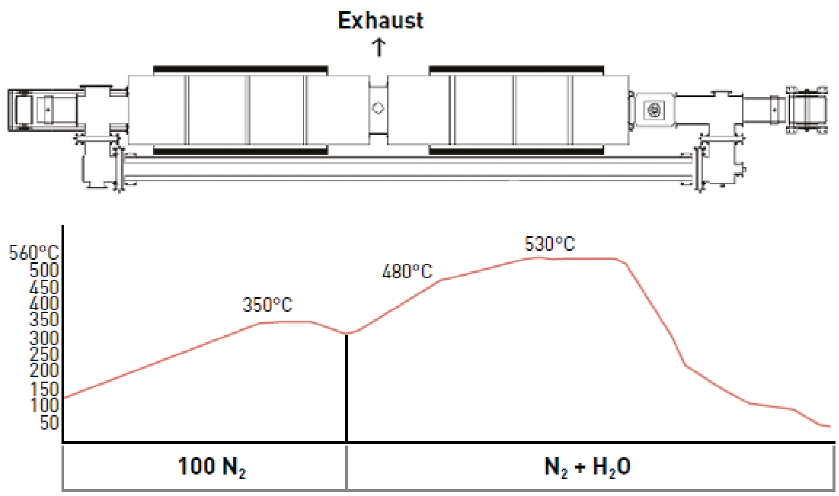

Fig. 8 An LGC furnace with features suitable for aluminium sintering and steam treatment would be suitable for the stress relaxation of soft magnetic alloy parts where required

Conclusion:

With the reduction in gas consumption and the range of applications made possible or made more efficient in Fluidtherm's Low Gas Consumption furnace, it is clear that this new technology could represent a key development for some segments of the metal powder-based manufacturing industry. The more cost efficient and versatile PM production becomes the easier it can compete with conventional manufacturing processes, and provide a wider range of solutions for a changing market.

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References

[1] *N. Gopinath et al, Continuous Case Hardening of PM Gears with Low Pressure Gas Quenching, presented at World PM 2016, Hamburg, Germany*
[2] *Th. Schubert et al, The importance of atmosphere and impurities on sintering behaviour of aluminium, presented at Euro PM 2005, Prague, Czech Republic*
[3] *N. Gopinath et al, Low temperature pusher furnaces for aluminium sintering & steam treatment, presented at the APMA 2017 Conference, Hsinchu, Taiwan.*

Low Temperature Pusher Furnaces Aluminium Sintering

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INTRODUCTION

Aluminium PM parts are used for the advantages they offer such as being non magnetic, having good mechanical & fatigue properties, low weight, corrosion resistance, high thermal & electrical conductivity, good ductility, excellent machinability & the possibility of employing several finishing processes, not the least being strengthening by heat treatment.
Sintering of aluminium PM Parts requires superior atmosphere integrity and very high temperature uniformity.
Low temperature pusher furnaces with entry and exit atmosphere lock chambers and an all metal muffle with gas re-circulation are ideally applicable for this demanding process for reasons described below.

ALUMINIUM SINTERING

The thermodynamically stable surface oxide layer on aluminum powder inhibits sintering and needs to be disrupted or removed. As this oxide reduction is not feasible with conventional furnace atmospheres MgO is added to partially reduce Al_2O_3 to form spinel $MgAl_2O_4$ which reaction causes Al_2O_3 film rupture exposing the underlying metal for neck formation (2).

Critical aspects of aluminium sintering are;

- Complete de-waxing before sintering to prevent hydrocarbons entering the sintering section.
- High temperature uniformity throughout the process (±3°C)
- High atmosphere purity, oxygen content not to exceed 5 ppm & dew point lower than - 60°C
- No hydrogen. Presence of hydrogen in the atmosphere even to a small extent prevents sintering shrinkage (3).

The layout of a low temperature pusher furnace to meet these requirements is given below (Fig. 5) & the photograph of the furnace is shown below (Fig. 6)

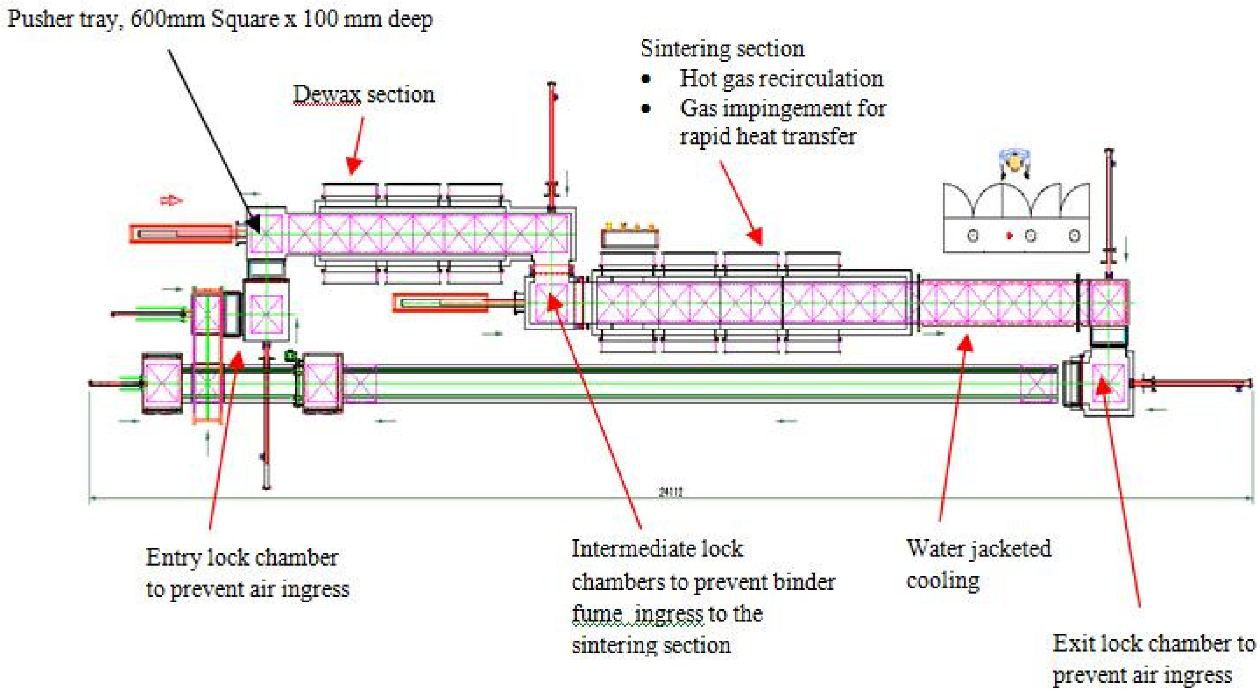

Fig.5 Furnace General Arrangement

Fig.6 Production scale furnace

After the furnace was commissioned a Temperature Uniformity Survey was conducted and the results were found satisfactory due to the design of the convection guide baffles which were employed (Fig 7).

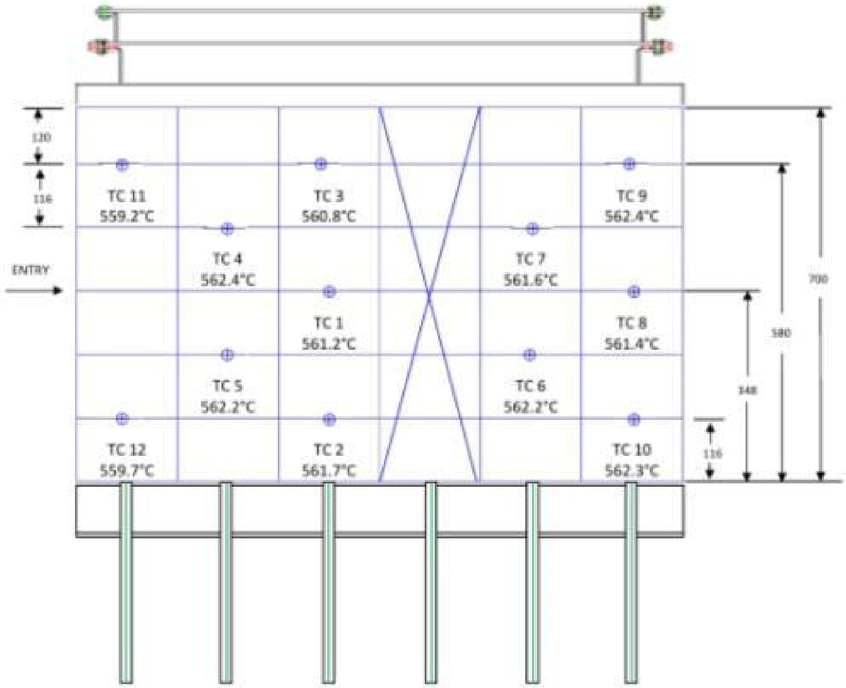

Fig. 7 TUS of the Al sintering furnace

Furnace zone set temp: 560°C
No of thermocouples/tray: 12
Max. Temperature recorded: 562.4°C
Min. temperature recorded: 559.7
Temperature Uniformity: +2.4°C / - 0.3°C
Nitrogen dew point inside the furnace: -60 to -65°C
Oxygen content inside the furnace: 3 to 13 PPM

Initial process trials on Alumix 231 from ECKA Granules were discouraging due to surface oxidation.

The furnace atmosphere was 100% Nitrogen with an impurity level of 13 ppm Oxygen, normally considered quite pure.

However the parts had an oxidized rim and were dark grey (Fig. 10). A photo micrograph (Fig. 9) showed that grain boundaries were present at the surface indicating lack of fusion. SEM analysis confirmed a higher level of oxygen on the surface compared to the core of the part.

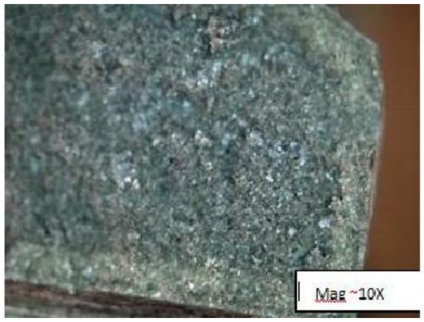

Fig.8

Photograph shows oxidised rim of a fractured sample

Fig.9

As a corrective measure the pressure of different sections of the furnace was analysed during events like door & pusher operation and parameters were altered to maintain a lower positive pressure in the atmosphere lock chambers. These measures caused a reduction of the oxygen impurity which fell from Max.13 ppm to Max. 5 ppm, as seen in a PPM oxygen analyser.

Subsequently processed parts were without surface oxidation (Fig. 10) and bright (Fig. 11). Physical & mechanical properties obtained are shown below (Table 2).

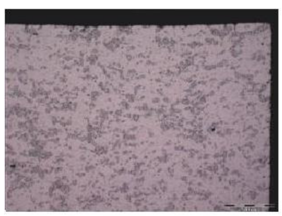

Fig.10

Microstructure shows absence of surface oxidation

Fig.11

Bright part

Table - 2 Properties of Processed parts

Sample no.	Properties as per manufacturer's catalogue		
	Shrinkage %	Tensile strength	Hardness HV0.5
	Typ. Value* 1.65%	Typ. Value* 170 Mpa	Typ. Value* 67 HV
	Properties achieved		
1.	1.53	169.01 MPa	67
2.	1.53	172.17 MPa	66
3.	1.51	170.59 MPa	66.5

After initial teething problems the use of a purpose designed low temperature pusher furnace proved satisfactory for the purpose of Aluminium sintering.

FLUIDTHERM

A NEW POST ALLOYING PROCESS "POWNITE" FOR IMPROVING MECHANICAL PROPERTIES OF SINTERED LOW ALLOY PARTS

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Presented at EPMA-2017, MILAN

ABSTRACT

A new post sintering gas alloying process is developed for improving material properties of low alloy sintered parts without quenching. This is achieved by a controlled level of nitrogen diffusion at a temperature between 590°C to 700°C and the resultant structure will be predominantly nitrogen rich austenite without rapid cooling depending upon the nitrogen concentration. This can be converted to further to get hard transformation products.

INTRODUCTION

A process technology has been developed at the Fluidtherm Technology Development Center where unalloyed PM parts are hardened by alloying the parts with Nitrogen at temperatures between 590°C to 720°C either as an extension of the sintering process or as a separate standalone process. Nitrogen is infused into the steel matrix to create a nitrogen rich austenite phase which is then converted by aging [1] at 200°C to 550°C into hard transformation products. This hardening process has most of the advantages of sinter hardening and in addition, reduces manufacturing cost further by reducing the use of expensive alloying elements and improving shape retention. As the authors could not find exactly such a process in literature it has been christened as the 'POWNITE Process' and is referred to it as such hereafter.

When nitrogen is diffused in low alloy PM parts, three reactions can take place depending upon nitrogen concentration [2] and temperature.

- Nitrogen enriched α (ferrite) phase
- Nitrogen enriched γ (austenite) phase
- Iron nitrides such as γ' and/or $\in$.

As nitrogen diffuses from the surface to core, the matrix structure is altered according to the concentration gradient.

This process differs from Nitriding type processes in that it is not a surface hardening technique which relies on the formation of a layer of iron nitride with or without the formation of supporting substrate layers as well as the processes of diffusion of Nitrogen without the formation of iron nitride. The positioning of conventional gas Nitriding, Ferritic & Austenitic Nitrocarburising are shown on the Lehrer diagram in red as against the positioning of the POWNITE process in the figure below. (Fig.1).

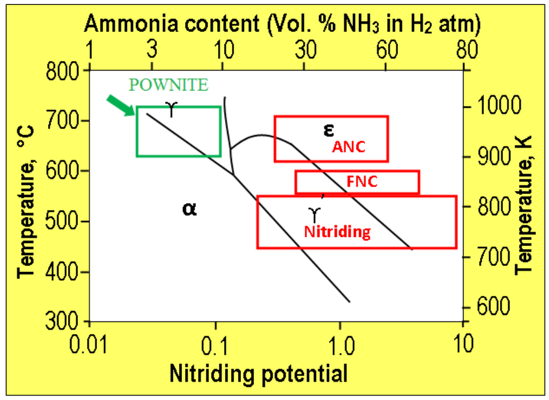

Fig. 1 The POWNITE process position in the Lehrer diagram

The depth, to which gas alloying occurs apart from process variables, is also in proportion to the part density. It is possible to strengthen the entire cross section of several standard PM parts with a density of up to 6.8 g/cc. The formation of brittle grain boundary iron nitrides is avoided. However, as an extension of the POWNITE process, surface iron nitrides can be created for improved wear resistance without embrittlement.

Experiments on PM bushes described in Table [1] based on a design of experiments were conducted in a purpose-built pusher furnace to optimize process parameters.

Table 1 Details of parts for POWNITE trials

Sintered Part Dimensions (Nom. mm)	18.00 OD, 12.00 ID and 18.6 Long			
Material	Fe-2% Cu-C			
Material code	A	B	C	D
Carbon %	0.5	0.5	0.8	0.9
Density (g/cc)	6.8	7.2	6.8	7.2

The process cycle adopted is shown in [Fig.2]

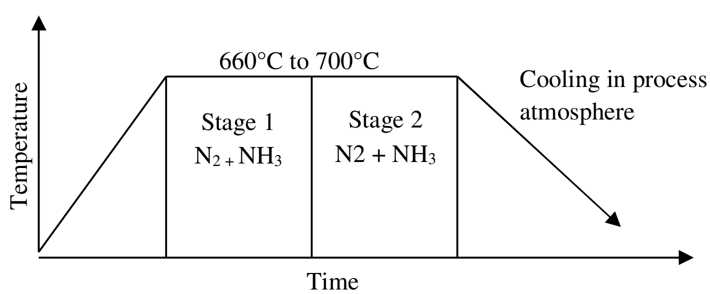

Fig.2 A typical POWNITE process cycle

Table 2 : Radial crushing strength (Mpa) of POWNITE processed parts compared with 'as sintered' and quench and tempered bushes

Material codes see (table 1)	A	B	C	D
As sintered	535.05	616.54	675.58	745.40
oil quenched & tempered	923.30	917.31	942.62	1025.87
POWNITE processed				
Exp. N5	829.64	693.33	531.32	601.15
Exp. N9	666.85	664.89	686.47	680.48
Exp. N3	705.10	841.41	762.96	909.57

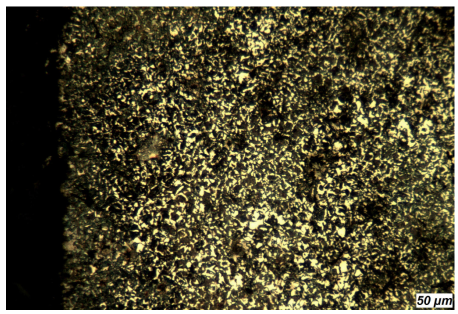

Fig.3 Surface structure showing Bainite with ferrite and without Iron Nitrides for N 5

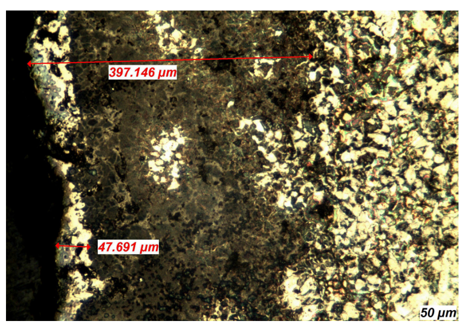

Fig.4 Surface structure showing a shallow layer of iron nitride with substrate of Bainite for N 9

Hardness profiles were taken on the processed samples to assess the surface to core hardness variation and compared with 'as sintered' and heat-treated bushes. (Fig 5 & 6)

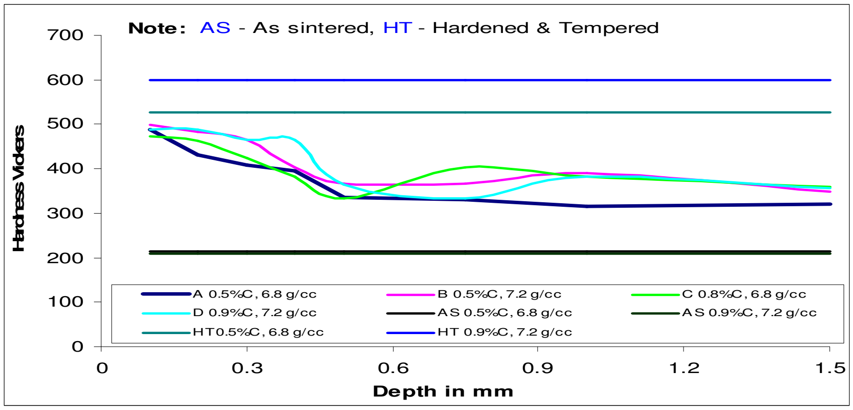

Fig. 5 – Hardness profile of Experiment N5

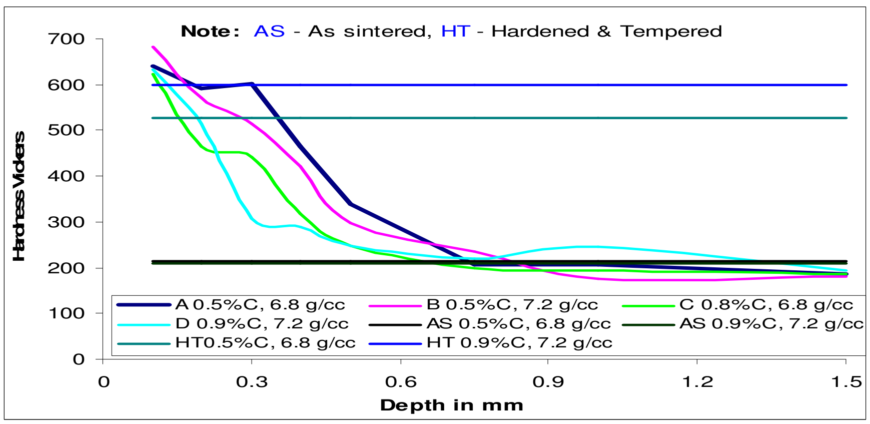

Fig. 6 – Hardness profile of Experiment N9

SEM - EDX analysis & N_2 profile

Nitrogen profile was analysed by SEM – EDX from surface to core at various locations (N5) had hard transformation products throughout the section thickness without any surface nitride layer. The core hardness is higher than the 'as sintered' bush but lower than the quench & tempered "control" sample had been processed for a higher surface nitrogen concentration for improved wear resistance (N9) and this shows higher surface hardness than the quench & tempered control sample.

The Nitrogen concentration profile, surface to core of the N5 & N9 samples is shown in [Fig. 7]

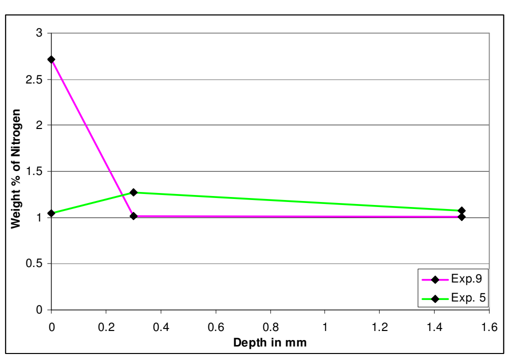

Fig. 7- Nitrogen weight percentage profile of N5 & N9

Microstructure of the Sprocket (POWNITE processed + aged at 450°C)

Fig 8 Material: C 0.5c, Cu 2.5%, Bal Fe

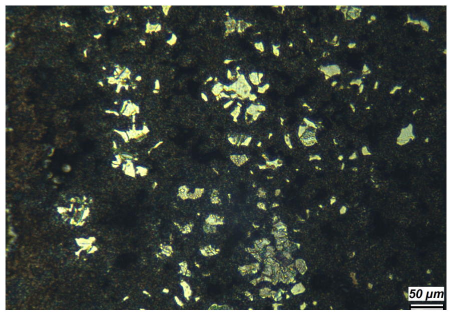

Fig. 9 Substrate microstructure - predominantly bainite
Surface hardness: 572 - 541 HV
Substrate (bainite) hardness: 412 - 520 HV

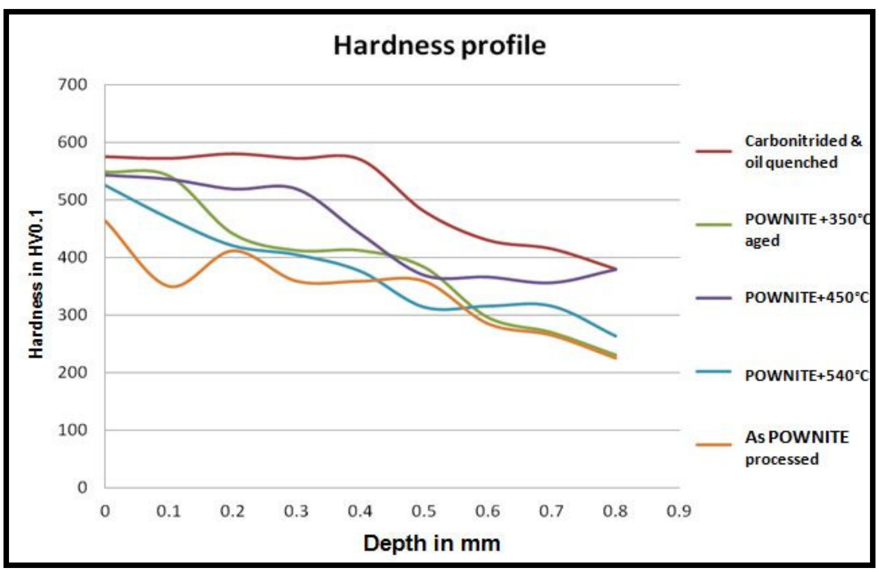

Fig. 10 Hardness profile from the surface of processed sprockets

SPECIFICATIONS	OBTAINED AFTER CARBONITRIDING	OBTAINED AFTER POWNITE PROCESSING AND AGEING AT 450°C
Surface Hardness 450HV Min	Surface Hardness 570HV	Surface Hardness 543HV
Case Depth 0.2 to 0.5 mm with at a cutoff point of 450HV	Case Depth 0.55mm with at a cutoff point of 450HV	Case Depth 0.38mm with at a cutoff point of 450HV
Core hardness 450HV Max	Core hardness 360HV	Core hardness 225HV

Table 3: Comparison: Carbonitrided and POWNITE processed sprockets

POWNITE PROCESS VS PRODUCT DENSITY

Sintered bushes ID-19.5mm, OD-29, L-26.5 with density 6.4, 6.6 and 7 gm/cc and material specification 0.5%C, 2%Cu and balance Iron were studied after POWNITE process and ageing at different temperatures to study the effect of density and process response. The results were also compared with oil quench and tempered bushes.

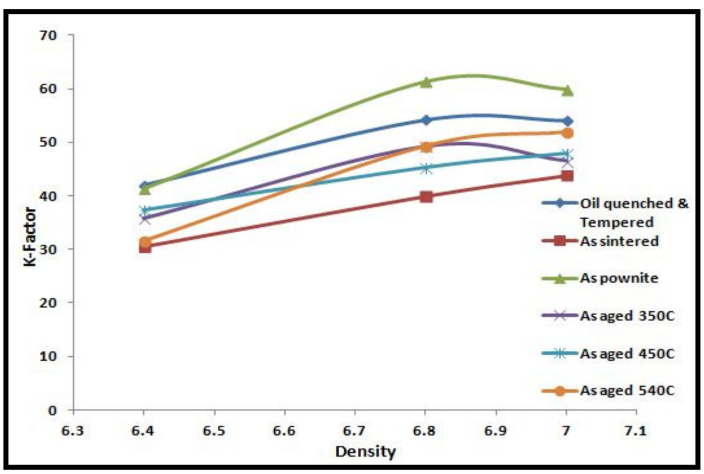

Fig. 11 Toughness parameter vs density

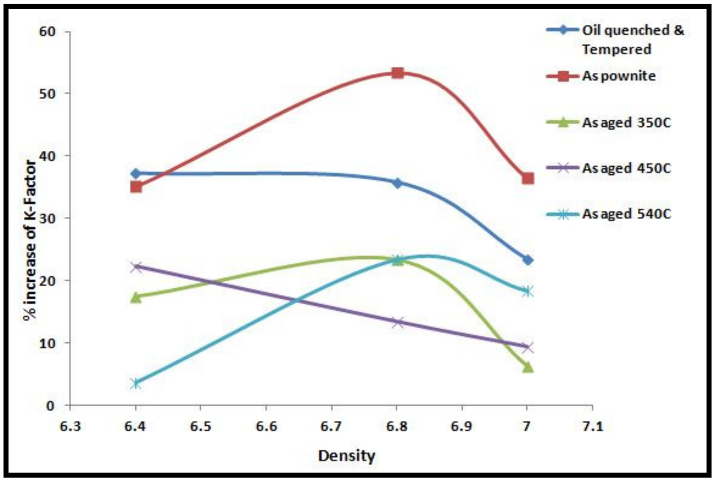

Fig. 12 % change as compared with sintered bush

Strength is higher in Pownite processed bushes comparing to as sintered condition at different densities.

Strength is higher than the as quenched and tempered products after pownite process without aging

As Sintered bushes can be replaced by Pownite processed lower section thickness bushes which can result in possibility of material saving up to 5 to 15%.

CONCLUSION

1. Gas alloying with appropriate process parameters enhances the mechanical properties of unalloyed PM parts. The strengthening is primarily due to the formation of hard transformation products and to a lesser extent, higher content of diffused Nitrogen in the matrix.
2. In addition to overall strengthening of PM parts, if the process is modified to provide a surface layer of iron nitrides the surface hardness achieved is comparable to the surface hardness achieved after conventional hardening and tempering and is therefore expected to provide similar if not superior wear properties.
3. The radial crushing strength of processed medium carbon parts is seen to be distinctly higher than the 'as sintered' part regardless of part density however it is lower than quench and tempered parts.
4. The radial crush strength comparison of the high carbon parts with the 'as sintered' parts shows mixed results.
5. Distortion performance is expected to be better than that obtained in hardening processes that involve rapid cooling (whether in conventional heat treatment or in sinter hardening) because gas alloying is performed at lower temperatures and the cooling rate is slow and relatively unstressed.
6. Strength increase after Pownite process shows possibility of reduction in section thickness that will result in material saving upto 5 to 15% for sintered bush bearing applications.

REFERENCES

1. Howard A. Ferguson, Metallurgical Consultant, Heat Treating of Powder Metallurgy Steels, ASM Handbook Volume 4. Pages 229 to 236.

2. Source: © 2003 ASM Handbook Vol 4 (Page #430 – 433). Practical Nitriding and Ferritic Nitrocarburizing (#06950G) by David Pye.

3. N. Gopinath & V. Raghunathan, "A New Post-Sintering Process to Improve the Mechanical Properties of PM parts by Gas Alloying ", PM 2014 World Congress, Orlando,