Powder Metallurgical Solution for a Complex Geometry Coupler Requiring High Dimensional Stability and Microstructural Uniformity through Heat Treatment

Craig Stringer, Andy Wright, and Pete Imbrogno
Atlas Pressed Metals, DuBois PA 15801 USA
craigstringer@atlaspressed.com

Abstract

Powder metallurgy (PM) is the fabrication process of compacting metal powders to shape and sintering these compacts to yield the final material's properties. The PM compaction process allows for complex geometries to be formed that would normally lead to long and expensive machining processes from wrought steels. Special alloy selection can allow for hardening of the microstructure during the sintering procedure. The sinter hardened (SH) alloys exhibit good mechanical properties along with good hardenability and dimensional stability and may be a suitable replacement for wrought steels where low distortion from heat treatment or microstructural control is required. In this study, it was found for a complex geometry coupler application, a SH alloy could successfully replace an austenitizing heat treatment process with a low carbon steel. The low carbon steel was found to have micro heterogeneities from heat treatment that lead to premature failure in the application. Dimensional distortion and production variance were also of concern with the low carbon steel. The SH material demonstrated acceptable physical properties, hardness and microstructural uniformity to solve the concerns associated with processing of the low carbon steel coupler. Post processing optimization also added to the life performance of the coupler by tailoring the final microstructure to mating components.

Introduction

Powder metallurgy processing is a net shape technology that provides outstanding shape accuracy and precision in high volume mass production with minimal or often no subsequent machining operations. For these reasons PM often ensures high cost efficiency and competitive pricing of structural component manufacturing as compared to other processing techniques. [1-5] Furthermore, structural PM components, being fabricated through conventional process route by compaction and sintering (frequently with complex geometry), quite often require secondary operations like machining, heat treatment (HT), plating, etc. in order to form the final properties. In particular, the HT of PM structural parts leads to sizable increases of strength, surface hardness, and wear resistance. A general summary of PM ultimate tensile properties is shown in Fig. 1 for several common materials families in the PM industry. From Fig 1. we can see the influence of a HT process on the various chemistries and the range of measurement for each with respect to process density, sintering conditions and resulting microstructures.

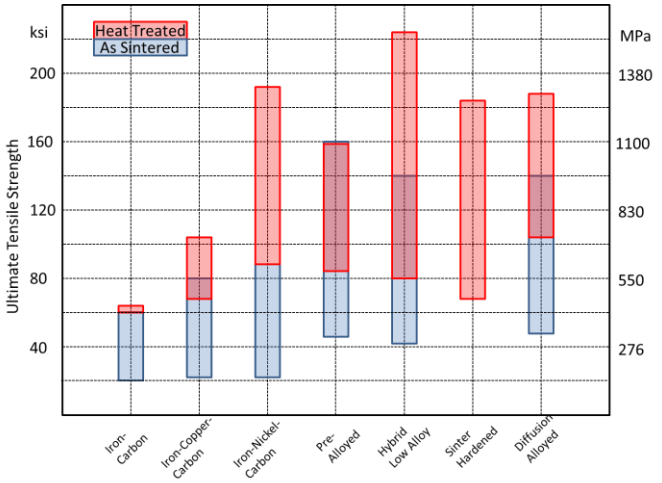


Figure 1. Chart summarizing ultimate tensile strength across several material families common in the PM industry with respect to as sintering properties and HT properties.

The heat treatment of steels is a complex process in which the component experiences drastic changes to microstructure and internal stresses as it is quenched through the various phase transformations. During this process, phenomena such as thermal gradients, phase transformation, and resultant internal stresses can contribute to distortion and/or inconsistencies in the resultant localized properties of the quench hardened components that can possibly lead to reliability concerns in an application. [6-8] Thus, by generating the proper phase composition within the steel part structure through application of various processing HT techniques, the desirable functional effect can be achieved. In applications requiring wear resistance and hardness, the dominance of the martensitic phase should be targeted, whereas demand for more toughness may require certain presence of bainitic phase, while for better ductility presence of pearlite might be desirable, etc. It is important to note that metallurgical phase transformations occur through every HT processing technique applied to components, regardless from either conventional wrought steels or from PM steels.

As it has been noted, the heat treatment of steels is complex and failures can result in an application from the process variations within the HT procedure. It has been estimated that when failure occurs from a HT procedure, roughly 20% of the problems relate to the heating process of the component and as

much as 80% can be related to the cooling or quenching process. [7] At the austenitization temperature, the steel is capable of dissolving up to 2 wt% of carbon in the face centered cubic (FCC) structure. As the steel cools, the ability to retain the carbon diminishes with the resultant microstructure dependent on the rate of cooling, e.g. pearlite (alternating layers of ferrite and cementite) for slow cooling and martensite (body centered tetragonal, BCT, structure) for fast cooling. [6] For an application that requires high wear characteristics it is important to cool at the appropriate rate to transform the microstructure to martensite. Martensite does not form until the M_s (martensite start temperature) is reached and 100% transformation occurs when the M_f (martensite finish temperature) is reached. The M_s and M_f temperatures are dependent on the alloy addition and especially the carbon content of the steel. [9] The rate at which the steel cools from the austenitizing temperature depends on several factors including the thermal conduction of the quenchant medium to pull heat away from the steel surface and also the heat flow inside the steel itself. Ideally, the rate of thermal conduction from the steel to the quenchant medium is infinite, i.e. the steel instantaneously reaches the temperature of the quenchant fluid. However, in reality this is not practical and largely dependent on the quenchant fluid itself, whether it be an air, water or oil quench. There are considered three stages of heat removal from the surface of the steel during a fluid quench operation, (1) vapor blanket stage, (2) nucleate boiling stage, and (3) liquid cooling stage. [10] The highest cooling rate during quenching occurs during the nucleate boiling stage. This follows the breakdown of the vapor blanket stage and can be facilitated through agitation of the quenchant fluid to ensure constant disruption of the vapor blanket. It has been established in the past that nonuniform quenching can be observed when agitation is not used and "hotspots" can result in heterogenous microstructures, inconsistent hardness and distortion. [10]

The resultant inconsistency in hardness can be correlated to the hardenability of the steel per the Jominy end quench test. [11-12] In this case, a decrease in hardness is measured as a function of distance (subsequent heat removal rate) from the quenched end of the steel bar. This is to say, if the quench process is not optimized, i.e. poor agitation, poor load placement or weight, or poor quenchant flow through complex component features, differing hardness values can result. In the case of distortion for instance, when the structural steel component is heated and subjected to rapid cooling, i.e. quenching, there is a rapid transformation from the austenitic phase, which has the highest density and lowest volume, to martensitic phase that has the lowest density but the biggest volume. The volume change ($\Delta V_{A\rightarrow M}$) of austenite volume ($V_A$) to martensite volume ($V_M$) can be linked with carbon content (wt.% C) of the steel with the empirical formula depicted in equation *(1):*

$$(\Delta V_{A\rightarrow M}) = 4.64 - 0.53 \times (\%C) \qquad (1)$$

For example, from equation *(1),* the volumetric change for a steel component that contains 0.8wt.% C is expected to expand up to ≈ 4.22 vol.% assuming 100% transformation from austenite to martensite, hereby causing a volumetric/dimensional change and possible distortion. [13]

The sinter hardening (SH) process in powder metallurgy allows for unique control of the hardened microstructure to minimize the aforementioned challenges of distortion and volumetric change within a quenching process. [14-16] The SH process provides a martensite microstructure through atmosphere quench inside a sintering furnace. PM components that are fabricated from the SH process are typically used in medium to high strength applications were precise dimensional control, high strength and wear resistance properties are required. The Metal Powder Industries Federation (MPIF), Standard 35, defines the SH family of materials with typical properties of the alloys showing the nature of the chemistry that allows for the phase transformations to occur with a critical cooling rate achieved in a sintering furnace. [14] The SH microstructure typically is fully martensite but regions of fine pearlite, bainite and nickel rich areas are also sometimes observed depending on furnace loading, alloy utilized, and thickness of the cross-sectional area of the component. Characteristically, the micro-indentation values achieved of the SH alloys are in the 600-800 HV0.1 range depending on the amount of carbon present in the alloy and can be tempered to desired levels with traditional tempering operations. Strengths of the SH family of materials range from 480-1100 MPa.

In this study, analysis is performed on a low carbon, carburize and quench coupler to determine optimized microstructure and micro-indentation hardness for durability. Powder metallurgy couplers utilizing SH family of materials were fabricated and tested to determine its viability to meet required durability and performance in the intended application.

Experimental Methods

In this study, a commercially available alloy based on MPIF standard 35 was selected as a potential conversion material for the low carbon, AISI 1018, heat treated coupler. The chosen material system was a FL-5305 specification with typical chemistry listed in Table 1 and was compacted to a density of 6.6 g/cm^3 for the coupler application.

Table 1. FL-5305 chemistry specification per the MPIF Std. 35

	Fe	C	Mo	Mn	Cr
Minimum weight %	Bal.	0.40	0.40	0.05	2.70
Maximum weight %	Bal.	0.60	0.60	0.30	3.30

The pre-alloyed FL-5305 powder was blended with a solid organic lubricant to allow for sufficient friction control under ejection of the component from the axial die and graphite for microstructural carbon. The compaction of the component occurred in an Osterwalder CA-NC 1600 CNC hydraulic compaction press capable of compaction tool motions to shape the complex features of the coupler. Sintering was completed in a production stainless steel mesh belt furnace capable of sintering temperatures to 1150°C and equipped with accelerated cooling units. An atmosphere of 5-10% hydrogen with a balance of nitrogen was used for all PM components in this study. The accelerated cooling unit allowed for microstructural transformation of the PM alloy to martensite with a controlled cooling rate of ~ 1.5-2.0 °C/s from 760°C to 205°C. Couplers were then tempered to achieve a desirable tempered martensite

microstructure to match mating components for durability purposes.

The coupler, shown in Fig. 2, is approximately 70 mm in length by 32 mm in diameter. The original coupler was fabricated with the 1018 low carbon steel that went through a carburize, quench and temper process. Details of this process are not known to the authors, but the process was completed in a bulk methodology with bulk loading of the couplers into a heat treatment basket.

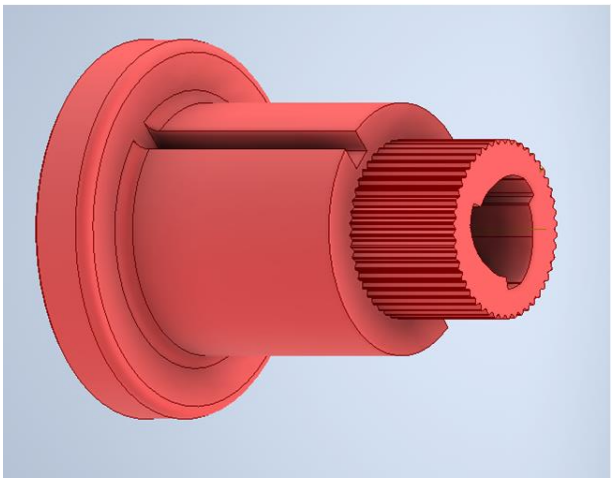
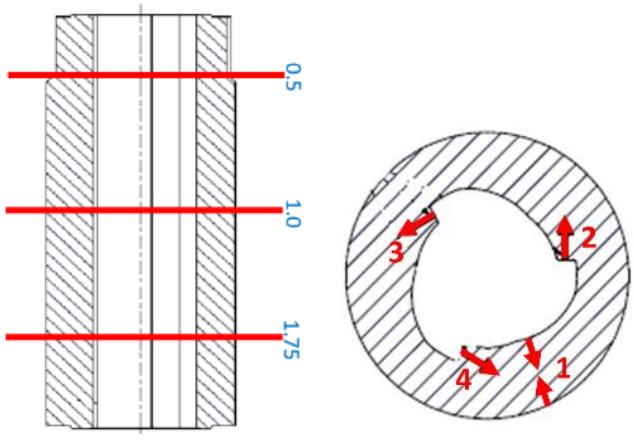


Figure 2. Solid model of coupler for study depicting OD and ID features for of the powder metallurgy process.

Evaluation of the low carbon coupler and PM coupler were identical in nature of microstructural determination, apparent hardness, and micro indentation phase hardness. The coupler was sectioned and evaluated as shown in Fig. 3(a). The coupler was sectioned at 3 locations along the body length and evaluated within the areas indicated in Fig. 3(b). These areas were identified as functional for the end use application of the coupler.



(a) (b)

Figure 3 (a) sectional view along body length and (b) radial cross section showing area of evaluation for microstructure and hardness. Number indicates identification of evaluation.

The microstructure was evaluated following best practice metallography techniques as described in the *MPIF Guide to PM Microstructures*. Micro indentation hardness was measured on a Struers Durascan machine utilizing a Vickers indenter with a force load of 100 grams. The samples were prepared and measured following MPIF std. 51 which requires 5 indentation measurements at each location.

Results and Discussion

Samples of the low carbon, carburized and quenched couplers initially received for evaluation were of a processing lot designation that resulted in mixed life test outcomes of premature failure (<50 hr) to expected life (~1000 hr). Samples were prepared for micro indentation measurement following an OD to core and ID to core depth profile as shown in location 1 of Fig. 3 (b). The depth- hardness profile is shown in Fig. 4 for 3 couplers that were sectioned at the 0.5 length of Fig. 3(a). The results indicated a broad range of measured values across the samples and at specific depths in relation to the OD or ID. As seen in Fig. 4, sample 2 exhibited a very high micro indentation hardness, in the range of 900 HV0.1 to 1000 HV0.1 at the surface, for both the OD and ID profile, while decreasing to a core hardness of ~ 400 HV0.1. It is interesting to note the difference in measured indentation hardness when compared to samples 1 and 3. Sample 2 had a more even hardness profile for the OD to core and ID to core while also exhibiting a higher overall measured indentation hardness profile. Samples 1 and 3 showed an unequal hardness profile from the OD to core and ID to core with the ID surface hardness being ~ 200 HV0.1 lower than the OD. The ID is the functional surface for the end application with a mating "key" component contacting the surface at the 0.5 section length of Fig. 3(a). The initial hypothesis was that the mixed life testing outcomes were a result of the mixed surface hardness values measured on the ID by micro indentation hardness. Both the OD and ID of all the measured samples had ~ 0.3 to 0.5 mm depth of martensite phase with the balance of the core having a pearlite/ferrite microstructure. The variance in the surface hardness profile was attributed to poor quench optimization with the bulk loading of the couplers during the heat treatment process.

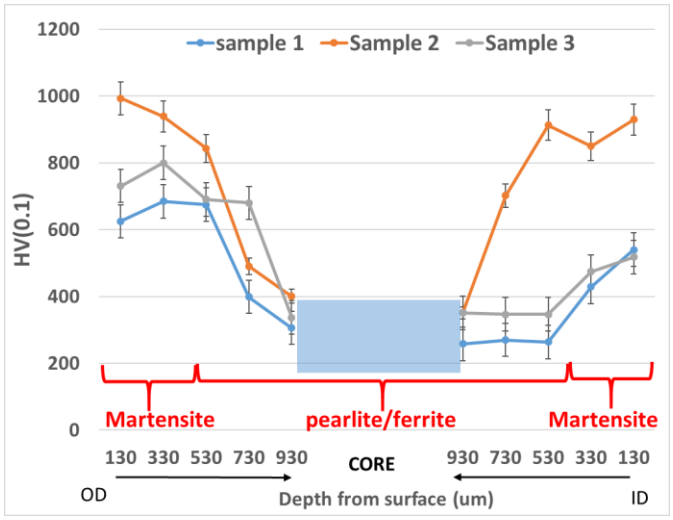


Figure 4. Measured micro indentation hardness (HV0.1) for samples showing mixed life test results. Martensite was observed in the surface region with the core of the coupler showing a pearlite/ferrite microstructure. Error bars represent the averaged range of measurement at each location.

Inconsistencies were also observed when measuring the micro indentation hardness in the other locations shown in Fig. 3(b) as is demonstrated in Fig 5(a-c).

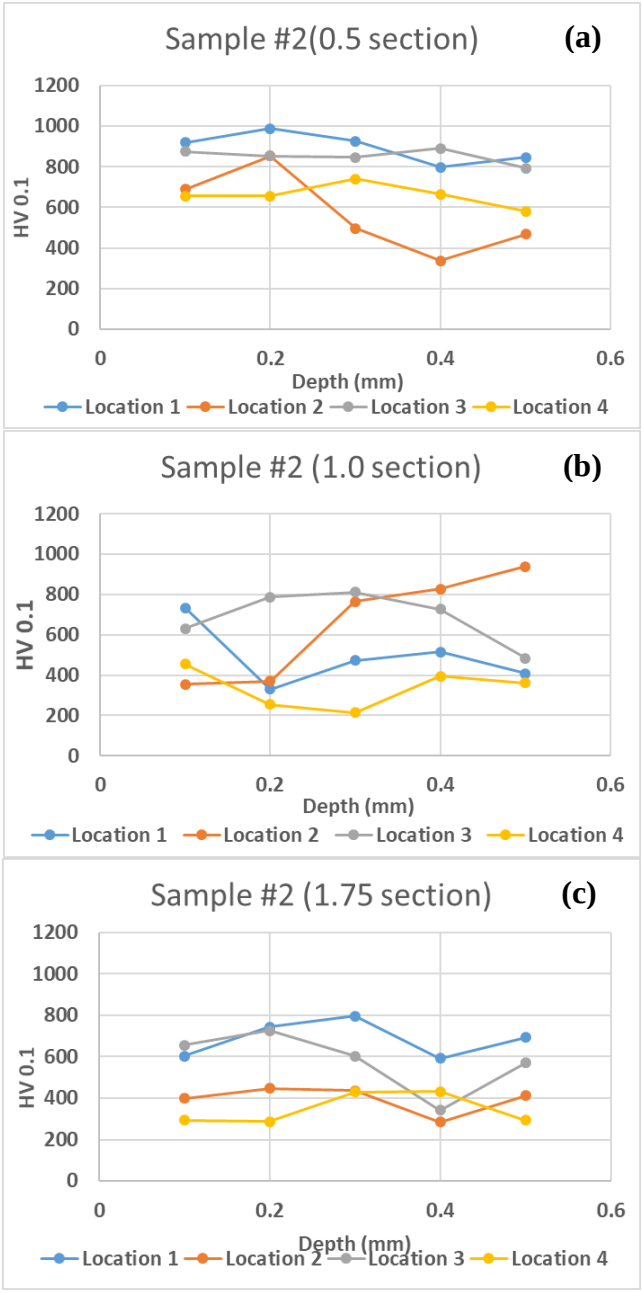


Figure 5. Measured micro indentation hardness profiles (HV0.1) at different sectional heights of the coupler as shown in Fig. 3(a).

What is notable in the Fig. 5 (a-c) profile curves is the variance of the surface hardness from one location to another within the same sectional height, i.e. from one side to another within the ID. It was observed on average to see ~ 300-350 HV0.1 difference when looking at the surface hardness from each location and up to a ~ 600 HV0.1 when looking at an extreme maximum and minimum across each location and sectional height. To some extent, the variance in hardness was not unexpected with the bulk loading of the couplers during heat treatment and the long, complex shape of the ID that the quenchant fluid must flow. It was determined by the original supplier of the couplers that the couplers may lie in any given orientation during the quench operation in relation to each other, i.e. 0º, 45º or 90º. Different contacts were also possible on the OD surface as the bulk loaded couplers would have regional contact variations depending on how each was oriented against one another. It is noted that stacking or racking the couplers could be an option for improved quench consistency and resulting micro-indentation hardness measurements. However, economic drivers and volume of pieces played a role in the end user searching for other methods for manufacture.

To determine the optimal ID microstructure and micro-indentation hardness, life testing was performed on several couplers and a post mortem evaluation was completed on >1000 hr life samples and <50 hr life samples. Fig. 6 shows the evaluation completed in a similar fashion to previous measures of both groups. It was identified, for optimal life of the assembly, that a coupler with an ID microstructure of martensite with a value of 450-550 HV0.1 micro indentation was ideal.

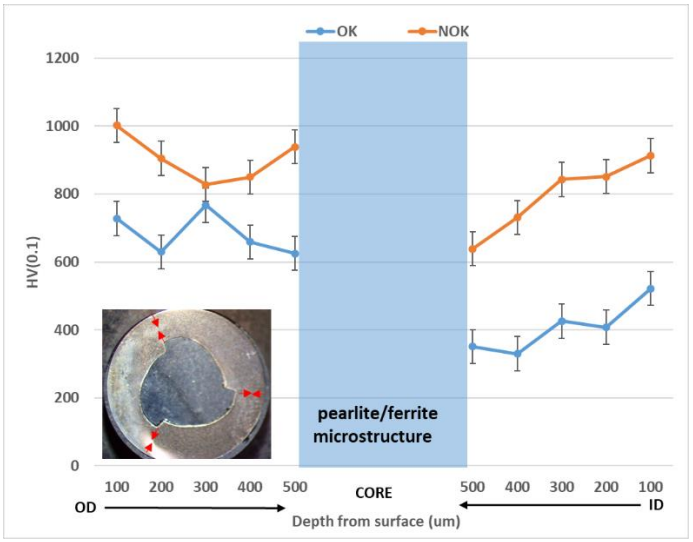


Figure 6. Chart of post mortem life cycle micro-indentation hardness for >1000 hr life (OK) vs <50 hr life (NOK) to determine optimum microstructure/hardness development. Error bars represent the averaged range of measurement at each location.

The OD structure was not functional to life performance but was measured for comparison purposes. The measured ID micro indentation hardness on the surface was found to be in the range of ~500 HV0.1 to match the material of the mating components. While the higher HV0.1 (~900 HV0.1) was found to diminish the life cycle performance with an unmatched hardness that would cause extreme wear on the "keyed" mating component. It should be noted, that while not measured in this study, there were two mechanisms related to poor life cycle performance: (1) extreme wear on the "keyed" component from a high coupler hardness and (2) extreme wear of the ID feature of the coupler from low coupler hardness. This is in alignment with variations observed in Fig. 5 (a-c) and the inconsistencies observed throughout the location/length hardness measurements.

The powder metallurgy coupler, fabricated from the MPIF FL-5305 material, has the ability to be sinter hardened through

furnace cooling as mentioned previously. This allows for direct control and uniformity of the microstructure and micro-indentation hardness throughout the length and location areas of the component. The component was compacted and sintered as described previously. The micro-indentation hardness measurement for the as sintered- furnace quenched samples had an average HV0.1 of 600-650. A tempering operation was used to draw the hardness down to an appropriate value per the optimized measurements of Fig. 6. The resulting micro-indentation values are shown in Fig. 7 for the cross-sectional designations explained in Fig. 3 (a). It was observed that uniform values for hardness were achieved both at the ID surface and through the cross-sectional thickness. The averaged values can be seen in Fig. 7 to fall between 535-580 HV0.1 which is ~ 1/6th the values obtained from the low carbon alloy, carburized, quenched and tempered couplers.

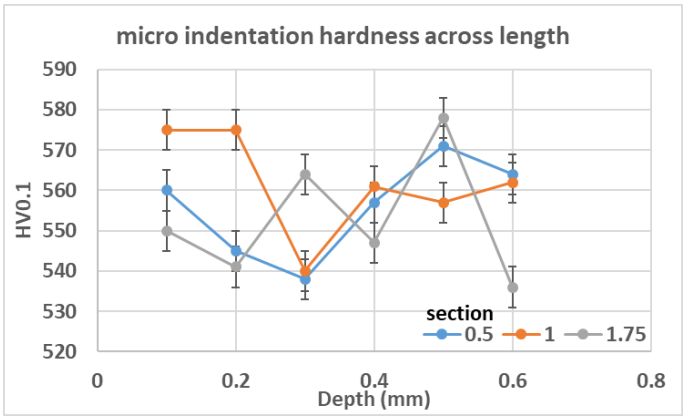


Figure 7. Micro-indentation hardness measurement of the PM coupler, FL-5305 as function of depth from the ID surface and sectional location. Error bars represent the averaged range of measurement at each location.

This also indicated a tempered martensite microstructure that has a tailored surface hardness that matches well with the "keyed" material of the mating component for durability and life cycle performance. The micro-indentation hardness was also measured along the sectional cross section level 0.5 in a similar fashion to that measured in Fig. 4 to determine the variance from location 2, 3, and 4 respectively, or from side to side of the complex ID features. From Fig. 8 we can see very little variance was measured to a depth of 0.5 mm for each location with an average hardness value of ~560 HV0.1 and a maximum range of 555 to 580 HV0.1. When compared to the low carbon, carburized, quenched and tempered coupler, we see an improvement from 200-300 HV0.1 difference to ~ 30 HV0.1 difference, or 1/10th the measured variance. The lot to lot production consistency was also measured across the first 5 production lots and can be seen in Fig. 9. The data is an average of the ID to core depth profile to 0.5 mm and shows a maximum variance of ~ 100 HV0.1 across all measurement. The average micro-indentation hardness for all production lots as a function is expressed as the black dotted line in Fig. 9 and shows very good consistency across the cross-sectional area. The PM coupler was measured for life performance in an identical manner to the low carbon coupler and all samples passed life expectation of >1000 hr cycle testing.

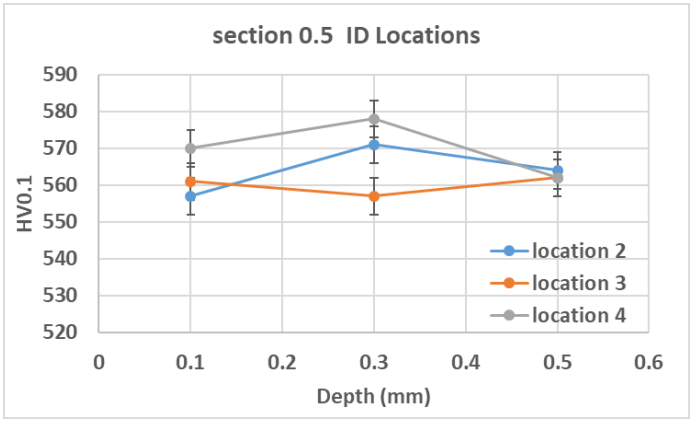


Figure 8. Micro-indentation measurement for locations 2, 3, and 4 respectively at a cross sectional height of 0.5 for the PM coupler. Very little variance between each location and to a depth of 0.5mm was observed. Error bars represent the averaged range of measurement at each location.

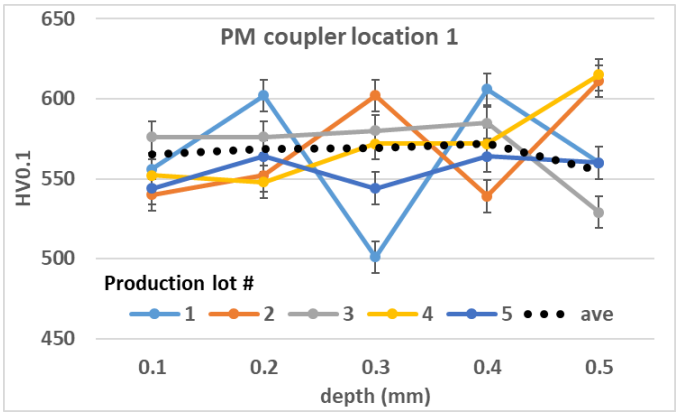


Figure 9. Lot to lot consistency of the PM couplers for the first 5 production lots completed. Each sample was measured at a cross sectional height of 0.5 and with the location 1, ID to core profile. Each data point is the averaged HV0.1 value through a depth of 0.5mm. Error bars represent the averaged range of measurement at each location.

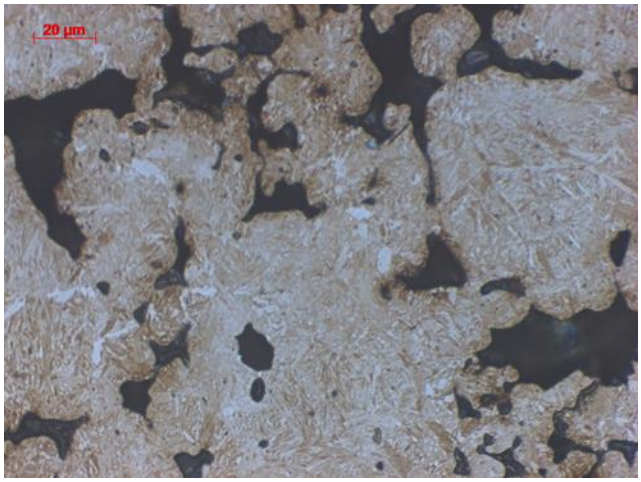


Figure 10. Typical microstructure of tempered martensite observed in the PM coupler. The black areas are porosity resulting from the compaction density of 6.6 g/cm^3.

Conclusions

In conclusion, it was shown that a powder metallurgy manufacturing method could be tailored to meet the requirement of a coupler, meeting durability and performance for the intended application. Due to a lack of a sufficient alloy content, the low carbon, carburized, quenched and tempered coupler was found to have inconsistencies in the microstructure and micro indentation hardness that lead to concerns for durability from premature wear on the assembled mating components. It was found that the variations were attributed to bulk HT processing of the couplers compounded by the complex ID features and the parts overall length leading to non-optimized quenchant fluid flow through the components. Powder metallurgy sinter hardening processing was demonstrated to minimize the variation in the microstructure/micro-indentation measurement at both the ID features along the length of coupler. Durability testing indicated acceptable performance from the PM coupler surviving >1000 hr life cycle compared to <50 hr for the low carbon coupler.

Acknowledgments

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References

[1] MPIF, *Powder Metallurgy Design Manual, 2nd Edition*, Metal Powder Industries Federation (Princeton, NJ, 1995) p 3-14.

[2] Randall M. German, *Powder Metallurgy and Particulate Materials Processing: The Processes, Materials, Products, Properties and Applications*, Metal Powder Industries Federation (Princeton, NJ, 2005) p 427-453.

[3] L.F. Pease and W.G. West, *Fundamentals of Powder Metallurgy*, Metal Powder Industries Federation (Princeton, NJ, 2002) p 1-10.

[4] F.V. Lenel, *Overview of Powder Metallurgy Products and Processing: A Lesson from Fundamentals and Applications of Powder Metallurgy*, ASM Int. Materials Engineering Institute, Course 0132 (ASM International, Materials Park, OH, 2002) p 1-20.

[5] S. Geroldinger, R. de Oro Colderon, C. Gierl-Mayer and H. Danninger. "Sinter Hardening PM Steels Prepared through Hybrid Alloying", HTM J. Heat Treatm. Mat. 76 (2021) 2, pp. 105-119

[6] Hoganas AB, *Hoganas Handbook No. 6: Metallography*, Hoganas AB, Sweden, 2015) p 300-301.

[7] L.C.F. Canale and G. E. Totten, "Steel Heat Treatment Failures due to Quenching," *Failure Analysis of Heat-Treated Steel Components*, (ASM International, Materials Park, OH, 2008) p. 255-284

[8] J. Dossett and G. E. Totten, "Heat Treatment Problems Associated with Design and Steel Selection," *ASM Handbook, Vol. 4D, Heat Treating of Iron and Steels*, (ASM International, Materials Park, OH, 2014) p. 1-28.

[9] G. Kraus, *Principles of Heat Treatment of Steel*, ASM International (Materials Park, OH, 1980) p. 35.

[10] F.C. Campbell, "Heat Treatment of Steel," *Elements of Metallurgy and Engineering Alloys*, ASM International (Materials Park, OH, 2008) p 177-199.

[11] ASTM Std. A255-20a, "Standard Test Methods for Determining Hardenability of Steel," *ASTM International*, (West Conshohocken, PA, 2020)

[12] ASTM Std. A304-20, "Standard Specification for Carbon and Alloy Steel Bars Subjected to End-Quench Hardenability Requirements," *ASTM International*, (West Conshohocken, PA, 2020)

[13] W.C. Leslie, "Iron and Its Dilute Substitutional Solid Solutions," *Metallurgical Transactions Vol. 3, 1972*, p 5-26.

[14] MPIF, *Metal Powder Industries Federation Standard 35, Materials Standards for PM Structural Parts*, Metal Powder Industries Federation (Princeton, NJ, 2018) p 26-27.

[15] F. Chagnon and Y. Trudel, "Optimizing Properties of PM Parts Through Selection of Proper Sinter Hardening Powder Grades," *Advances in Powder Metallurgy and Particulate Materials PM2TEC 2003, part 5*, p 77-88.

[16] F. Hanejko, A. Taylor and A. Rawlings, "Advanced Sinter-Hardening Materials and Practices," *Advances in Powder Metallurgy and Particulate Materials PM2TEC 2002, part 13*, p 102-127.