Dimensional Control in Powder Metal Parts

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Abstract

Powder metallurgy is a netshape process and hence an understanding what controls dimensional change is important. The alloying elements such as C, Mo, Ni, Mn, Cr and Cu will increase the hardenability of steel. The amount of prealloyed elements in the base iron will affect the transformation products found after sintering. The cooling rate, prealloyed elements and admixed additions will determine the final microstructure and mechanical properties in the sintered compact. Investigations on pure iron powder alloyed with Cu and graphite, prealloyed Mo alloyed with graphite reveal that better dimensional control is achieved using0.3% prealloyed Mo powder in heat treated condition. Four Sinter hardening grades were evaluated and results suggest Ancorsteel 4300 is most stable and provides a good dimensional precision.

Key words: Dimensional precision, prealloyed, sinter hardening ,tempering

Introduction:

Powder Metallurgy is a green process with over 97%material utilization and lowest energy per kilogram of part produced compared to other metal working processes(1).

The major advantage of powder metal parts is that it is produced in net shape configuration with very little machining. The technology is well accepted by automotive companies globally and applications continue to increase. Dimensional precision although good but efforts continue to improve the precision of powder metal parts.

Understanding the factors involved in improving precision are being addressed by the industry globally.

Hoeganaes developed number of dimensionally stable materials and in this paper we review the work done in the past in these areas.

FC-0208 is used extensively in the industry because of the ease of manufacturing. Dimensional precision of parts made from these powders needs improvement , Cu diffusion in iron is often controlled by the graphite diffusion which is controlled by the heat up rate to sintering temperature. Although the elemental copper addition melts during initial sintering, complete homogenization of the copper is not achieved at conventional sintering temperatures and sintering times. This lack of microstructural homogeneity coupled with the potential for large pores can often lead to reduced strength and greater dimensional variability during sintering and subsequent heat treatments In this study we compared a 0.3%pralloyed Mo powder with FC-0208 premixes.(2)

The demand for higher mechanical properties and lower cost for increasing powder metal penetration in the automotive applications resulted in the development of a number of sinter hardening grades(3). We compare four sinterhardening grades and studied dimensional precision after various heat treatments.

Experimental:

Comparison of FC-0208 to 0.3% molybdenum with 0.8% graphite

Four premixes were prepared as shown in Table 1. The mixes containing atomized Acrawax C were conventional premixes prepared via double cone blending. The premixes containing the warm die lubricant (AncorMax 200™) were prepared via a proprietary premixing process.

Table 1.Composition of Premixes

Mix	Base Iron	% Copper	% Graphite	% Lube & Type
1	Unalloyed iron powder	2	0.8	0.75% Acrawax
2	Unalloyed iron powder	2	0.8	0.4% warm die lubricant
3	Prealloyed 0.3% Molybdenum	NA	0.8	0.75% Acrawax
4	Prealloyed 0.3% Molybdenum		0.8	0.4% warm die lubricant

The following test specimens were compacted from the four premixes listed in Table 1.

Compressibility test specimens at 415, 550, and 690 MPa (30, 40, and 50 tsi).
For the warm-die premixes additional compressibility data was generated at 830 MPa (60 tsi). The conventional premixes were compacted in dies at room temperature; whereas, the AncorMax 200 premixes were compacted at 93 °C (200 °F), no powder preheating.

Transverse rupture strength bars for both as-sintered and heat treated strength at the various compaction pressures.

MPIF standard dog-bone type tensile specimens for both as sintered and heat- treat conditions (MPIF standard 10).

Sintering of all samples was done at 1120 °C (2050 °F) in a continuous belt furnace in an atmosphere of 90% nitrogen / 10% hydrogen for ~20 minutes at temperature; the cooling rate after sintering was ~0.6 °C per second (no accelerated cooling). All samples were sintered on ceramic trays. Heat-treated samples were re-austenitized at 815 °C (1500 °F) in a 75% hydrogen / 25% nitrogen atmosphere for 1 hour at temperature. They were oil quenched in 65 °C (150 °F) oil and subsequently tempered at 205 °C (400 °F) for 1 hour in a nitrogen atmosphere.

These premixes were utilized to evaluate the dimensional change variability during the heat-treat response of a prototype gear. The gear geometry used is shown in Fig 1. Approximately 1,000 gears were made from each premix, the two conventional double- cone premixes were run in unheated tooling with a target part density of 7.1 g/cm³ and the warm-die lubricant premixes were run in a die heated to 93 °C without top punch heating and a target part density of ~7.1 g/cm³. The compacted gears were sintered at 1120 °C (2050 °F) for 20 minutes at temperature in a 90% nitrogen / 10% hydrogen atmosphere in a 6-inch ceramic belt furnace without accelerated cooling. From these sintered gears 50 were randomly chosen and sent to a commercial heat-treat facility for a quenching and tempering operation. The heat treat cycle used was to austenitize at 870 °C (1600 °C) for 1 hour in a 0.8% carbon atmosphere followed by oil quenching and tempering. From these 50 heat-treated gears ~20 were then measured for part-to-part dimensional variation via a measurement over wire (MOW) of the gear form.

Fig 1: Spur gear produced to evaluate part-to-part consistency in the heat- treated condition

Gear dimensions were Major O.D 28,3 mm, major I.D.21.6mm,pitch diameter 24.5 mm, pressure angle 20°,I.D. 9.5 mm, number of teeth 16,module 1.66.

Property comparison of FC-0208 vs. 0.3% prealloyed molybdenum steel with 0.8% graphite:

Fig 2 presents the compressibility curves of the four materials listed in Table 1. As previously noted, compaction of the Acrawax containing premixes took place in unheated dies at room temperature; whereas, compaction conditions of the warm-die powder premixes was in dies heated to 93 °C (200 °F) without any prior powder heating. The compressibility of the prealloyed 0.3% molybdenum steel is identical to the FC-0208 material at both room temperature and at die temperatures of 93 °C. Utilizing warm die processing results in a 0.05 to 0.10-g/cm³ increase in green density relative to conventional lubricants for both material options. The optimal benefit with warm-die compaction is observed at compaction pressures greater than 550 MPa (40 tsi).
Although not shown, warm die processing also promotes higher green strength with reduced green expansion and reduced ejection forces.

The as-sintered and heat-treated transverse rupture strengths as a function of sintered density for the FC0208 and 0.3% molybdenum prealloy with 0.8% added graphite are presented in Fig 3. Warm-die processing did not affect the sintered or heat-treated strength. As anticipated, in the as-sintered condition, the FC-0208 material possesses higher strengths when compared to the prealloyed 0.3% molybdenum material.
However, once heat-treated the strengths of the two materials are nearly equivalent.

As-sintered tensile properties and heat-treated tensile properties for the 0.8% graphite additions are presented in Table 3. Similar to the trend observed with transverse rupture strength testing, the as-sintered yield and ultimate tensile strengths of the FC-0208 are greater than the prealloyed 0.3% molybdenum steel. Elongation values are nearly the same. Also presented in Table 3 are the ultimate tensile strengths of the FC-0208 vs. the 0.3% molybdenum steel in the quenched and tempered condition. Similar to what was found in TRS testing, in the heat-treated condition the prealloyed 0.3% molybdenum steel with 0.8% graphite and heat-treated FC-0208 have nearly identical ultimate tensile strengths. The data presented Table 3 represent compaction at 415, 550, and 690 MPa. The prealloyed 0.3% molybdenum material has lower growth relative to the FC-0208, thus the sintered densities at the same compaction pressure are higher for the prealloyed molybdenum material.

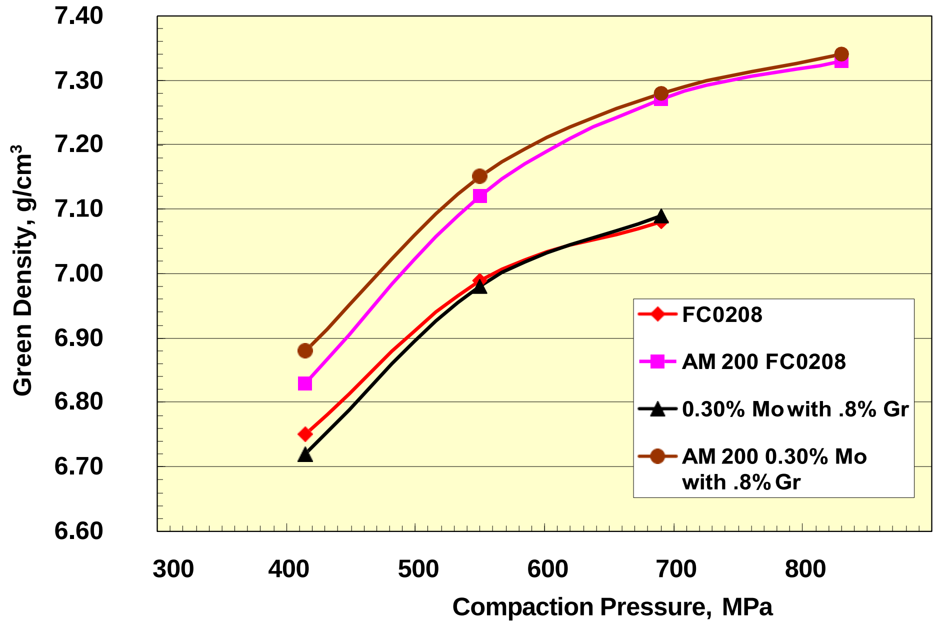

Fig 2: Compressibility of the four premixes

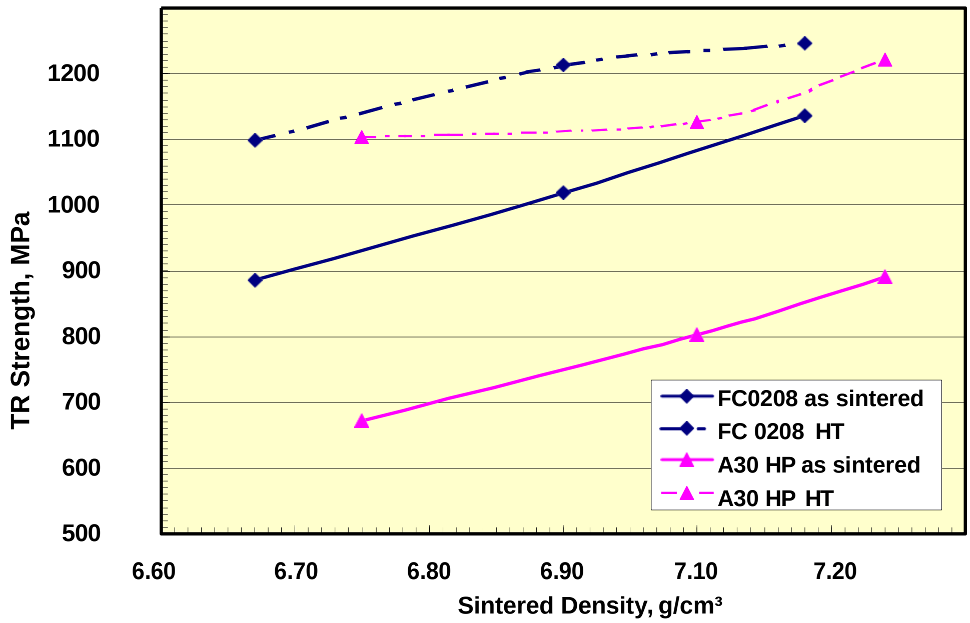

Fig 3 As-sintered and heat-treated transverse rupture strength.

Table 3: Summary of Tensile Properties of FC0208 and Prealloyed 0.3% Molybdenum Steel

Material	Sintered Density, g/cm^3	Heat Treated	0.2% YS (MPa)	UTS, (MPa)	Total Elongation (%)	Hardness, (HRA)
	6.67		393	470	1.4	47

FC0208	6.90	No	448	564	1.8	50
	7.04		467	597	1.9	53
	6.67	Yes	NA	672	<1.0	64
	6.90			771		69
	7.04			870		69
0.30% Mo Pre-alloy with 0.80% Graphite	6.75	No	293	334	1.2	40
	7.00		333	415	1.6	45
	7.14		359	435	1.7	47
	6.75	Yes	NA	705	<1.0	64
	7.00			805		69
	7.14			855		71

Results of heating treating of the gear shown in Fig 2.

Prototype gears were produced using the four materials discussed in part one of this study. They were made on a Dorst 140 metric ton compaction press at a rate of 10 per minute. The gears were sintered in a continuous belt sintering furnace at 1120 °C (2050°F) in a 90% nitrogen / 10% hydrogen atmosphere with a time at temperature of ~20 minutes. After sintering approximately 50 gears from each material grouping were heat- treated by quenching and tempering utilizing a commercial heat-treat cycle for FC0208. After quenching, the gears were then tempered at 205 ° C (400 °F). The gears were checked for dimensional variation using 3.162 mm (0.1245-inch) diameter pins.Measurements were taken at four locations per gear starting with the front of the gear (as compacted) and then measuring three additional locations around the outside diameter. The results of the measurement over wires (MOW) are presented in table 4.

Table 4 Dimensional Variations of Heat Treated Gears

Material	**Av. MOW Inches (mm)**	**Max MOW Inches (mm)**	**Min MOW Inches (mm)**	**Max – Min Inches (mm)**	**St Dev MOW**
FC-0208 (regular premix)	1.2096 (30.72)	1.2118 (30.78)	1.2075 (30.67)	0.0043 (0.11)	0.0013 (0.03)
0.3% Mo with 0.8% gr (regular premix)	1.2090 (30.71)	1.2096 (30.72)	1.2081 (30.69)	0.0015 (0.04)	0.0003 (0.01)
FC-0208 (bonded premix	1.2099 (30.73)	1.2117 (30.78)	1.2082 (30.69)	0.0036 (0.09)	0.0008 (0.02)
0.3% Mo with 0.8% gr (bonded premix)	1.2084 (30.69)	1.2089 (30.71)	1.2075 (30.67)	0.0014 (0.04)	0.0003 (0.01)

Table 5
Apparent Hardness Values of Gears in the Heat-Treated Condition

Material	Sintered Density, g/cm³	Average Hardness, HRA	Range of Hardness, HRA
FC-0208 (regular premix)	7.01	68.1	64.0 / 71.4
0.3-0% Mo with 0.8% gr (regular premix)	7.05	70.4	67.1 / 72.7
FC-0208 (bonded premix)	7.04	69.3	64.2 / 73.5
0.3% Mo with 0.8% gr (bonded premix)	7.10	71.0	67.5 / 73.5

The data presented in Table 4 clearly demonstrate the reduced scatter realized with the prealloyed 0.3% molybdenum steel relative to a standard FC-0208 material. The absolute magnitude of the MOW is different for the two material combinations; this is a result of the absolute DC differences between an FC-0208 and a 0.3% molybdenum prealloyed steel with 0.8% graphite. Thus, it can be clearly seen that a molybdenum steel gives greater dimensional stability in the heat-treated condition. No green or sintered gears were measured because the focus of this report was to investigate heat- treated results.

For the FC-0208 material, the binder treated warm-die powder gave a reduced overall scatter as measured by the maximum measurement minus the minimum measurement and also the standard deviation of the measurement differences. Thus, the binder treatment does result in reduced part-to-part scatter even for the copper-containing steel. No difference in dimensional variation was observed for the binder treated 0.3% molybdenum steel.

Apparent hardness data collected on the gears are presented in Table 5. The gears were initially compacted to a 7.1 g/cm³ green density; the densities given in the table are heat-treated densities. Another benefit of the low alloy steel powder is the greater uniformity of heat-treated hardness as measured by the range of hardness readings.

One potential benefit of eliminating the copper may be a reduced tendency for cracking during induction hardening. Induction hardening of copper containing PM steels has shown sensitivity to cracking. A possible explanation is the non-uniform microstructure inherent with copper containing PM steels. Eliminating the copper would eliminate one potential for crack initiation during induction hardening of the PM materials.

Sinterhardeanble Grades:

Austenite is the highest density phase in steel, followed by ferrite plus carbide, and finally martensite, which is the lowest density phase. This decrease in density results in a corresponding increase in compact size, and martensite formation leads to the largest growth resulting from microstructural changes. Length changes in pore free materials can reach 1.4% upon transformation from austenite to martensite . Tempering of martensite leads to a ferrite and carbide structure, which has a higher density. The post sintering operation of tempering should therefore decrease the dimensional change of a martensitic sintered compact. However, retained austenite is not a stable phase at temperatures below the eutectoid temperature. A reduction in temperature below room temperature will continue to drive the martensite transformation, while an increase above ambient temperature will encourage bainite formation. Transformation of retained austenite to either martensite or bainite will result in compact growth. The purpose of this study is to investigate the effects of post-sinter processing on the dimensional change and microstructure of sinterhardened steels.

EXPERIMENTAL PROCEDURE

The alloys studied were FLC2-4808 (Ancorsteel* 737 + 2wt% Cu + graphite), FLNC-4408 (Ancorsteel 85HP + 2wt% Ni + 1.5wt% Cu + graphite), FLC-4608 (Ancorsteel 4600 + 2wt% Cu + graphite) and Ancorsteel 4300 + graphite. The compositions are listed below in Table 6. The mixes were compacted into standard TRS, dogbone tensile, Charpy impact and dilatometry test specimens at a compaction pressure of 690 MPa (50 tsi) at room temperature. The samples were sintered in a 90% nitrogen – 10% hydrogen atmosphere at 1120 °C and accelerated cooling was used to achieve a cooling rate of 1.6 °C/sec (2.8 °F/sec) between part temperatures of 650 °C (1200 °F) and 315 °C (600 °F). Tempering was carried out in a nitrogen purged furnace at 205 °C (400 °F) for the majority of test conditions. After sintering, samples were subjected to a variety of tempering (T) and liquid nitrogen quench (LNQ) operations: 1. as- sintered, 2. LNQ, 3. T/1hr, 4. LNQ + T/1hr, 5. T/1hr, cool to room temperature + T/1hr .

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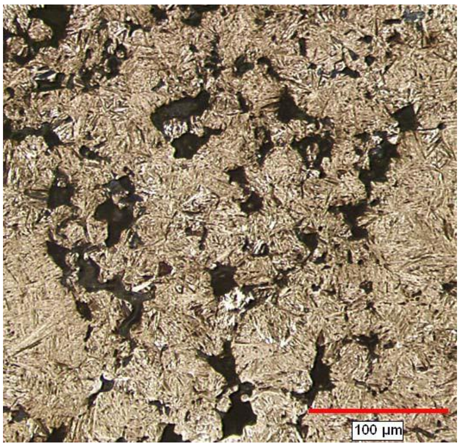
Table 6. Nominal compositions (in wt.%) of the alloys studied, balance Fe.

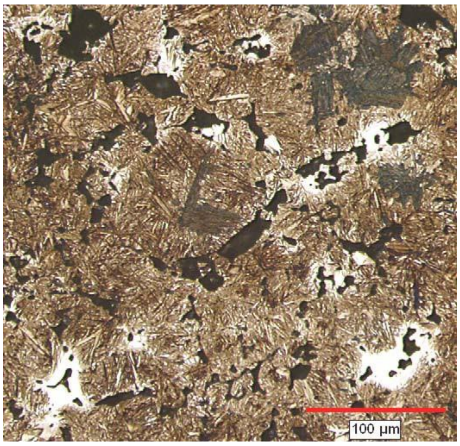
Alloy #	**Designation**	**Ni**	**Mo**	**Mn**	**Cu**	**Cr**	**Si**	**C**
1	**FLC2-4808**	**1.4**	**1.2**	**0.4**	**2**	**-**	**-**	**0.8**
2	**FLNC-4408**	**2.0**	**0.8**	**0.1**	**1.5**	**-**	**-**	**0.8**
3	**FLC-4608**	**1.8**	**0.5**	**0.1**	**2**	**-**	**-**	**0.8**
4	**Ancorsteel 4300**	**1.0**	**0.8**	**0.1**	**-**	**1.0**	**0.6**	**0.6**

RESULTS

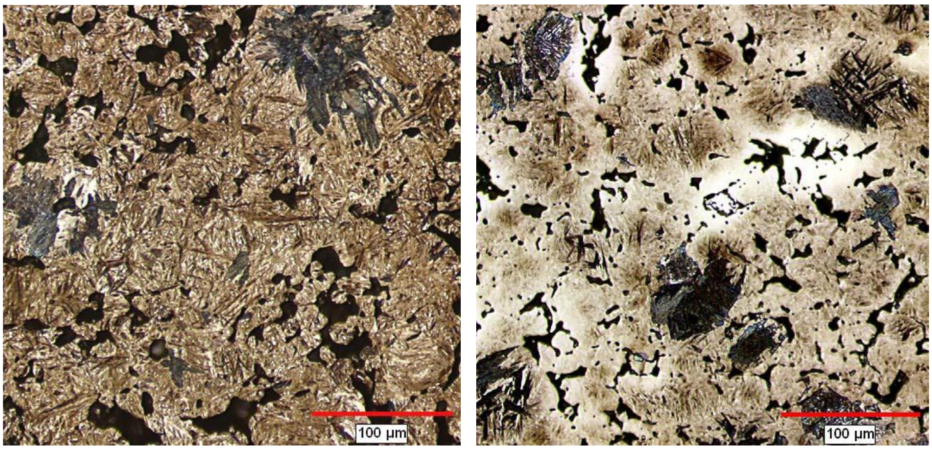
The as-sintered microstructure of the four sinter-hardenable alloys is shown in Fig 4. Alloy 1 (FLC2- 4808) is fully martensitic, while the other three alloys are predominately martensitic with a small percentage of bainitic regions. Nickel-rich regions can be found in the FLNC-4408 (Alloy 2) microstructure. Alloy 1 has the highest hardenability of the four alloys and can be cooled at a relatively slow rate while still maintaining a fully martensitic microstructure. The other three alloys have lower hardenability, and cooling rate of the sintered compact will play an important role in the final microstructure. One should be aware that the amount of martensite will play a role in the final dimensions of the part and how it responds to tempering. Therefore cooling rate must be well controlled to develop reproducible microstructures and dimensions.



Alloy 1 : FLC2-4408



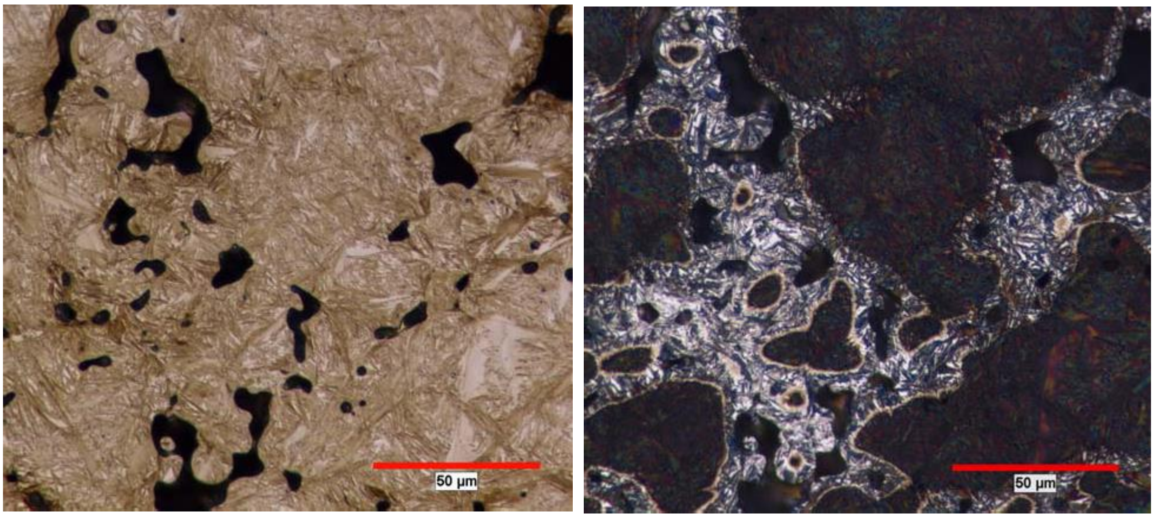
Alloy 2 : FLNC-4408



Alloy 3 : FLC-4608 Alloy 4 : Ancorsteel 4300

Fig 4. As-sintered microstructures of sinter-hardened Alloys 1-4 (2% Nital/4%Picral Etch).

Alloys high in carbon and copper with martensitic microstructures, such as Alloys 1-3, often contain a large fraction of retained austenite. Fig 5a shows the martensitic microstructure of Alloy 1 using a typical etchant, whereas Fig 5(b) shows the same field etched to reveal retained austenite (white). The majority of the retained austenite is directly associated with copper rich regions. Notice the retained austenite is present surrounding the pore network where copper concentrations are highest. Earlier work on this alloy has shown retained austenite levels as high as 10% . Given that the retained austenite is not uniformly distributed, local concentrations of austenite near the pore network will be much higher than the average value reported in the earlier work. Dimensional control of these sinter-hardened structures will be governed by not only tempering of martensite but also the transformation of retained austenite.



(a) 2% Nital / 4% Picral (b) as (a), then $Na_2S_2O_5$ in Water

Fig 5. Microstructure of the sinter-hardened Alloy 1 (FLC2-4808) at 1.6 °C/sec etched to reveal (a) martensite and (b) retained austenite (white) in Cu-rich regions (same field).

The early stages of tempering involve 3 phenomena: 1) rapid stress-relieve and diffusion of C to dislocations at relatively low temperature, 2) transformation of retained austenite to bainite, and 3) further C diffusion out of the martensite associated with the formation of carbides above 200 °C (400 °F).The first and third phenomena involve a reduction in length, as the body center tetragonal (bct) structure of martensite changes to bcc ferrite and carbide. The second produces a length increase, as the high density austenite phase transforms to the lower density bainite.

The dimensional change of Alloys 1 - 4 for the various thermal treatments is given in Table 7 . The results have been normalized to an as-sintered dimensional change of zero. Alloys 1 – 3 have significant levels of retained austenite, resulting in significant growth (+0.13% to +0.14%) with the LNQ. Tempering of the martensite results in shrinkage, as the bct structure of martensite converts to a bcc ferrite and carbide structure. Shrinkage results are seen in Table 7, where the pre and post temper condition of the as-sintered and LNQ samples are compared. More shrinkage occurs during tempering of the LNQ sample, as there is no growth contribution from conversion of retained austenite. It is interesting to note that the growth of Alloys 1-3 upon LNQ was similar, but the shrinkage during tempering is consistently lower for Alloy 2. The double tempered samples and the samples show growth relative to the single tempered samples as the retained austenite transforms to bainite. Samples only grew between 0.05% and 0.08% relative to the tempered condition, whereas the LNQ samples grew between +0.13% and +0.14 %

Table 7. Dimensional change (%) of post sinter operations normalized to the as-sintered length. Alloy 1 is FLC2-4808, Alloy 2 is FLNC-4408, Alloy 3 is FLC-4608 and Alloy 4 is Ancorsteel 4300 with 0.6% graphite.

Alloy #	1. As-Sinter	2. LNQ	3. T	4. LNQ + T	5. T + T
1	0	+0.13	-0.10	+0.01	-0.08
2	0	+0.13	-0.06	+0.04	-0.04
3	0	+0.14	-0.10	+0.01	-0.07
4	0	+0.06	-0.03	+0.02	-0.03

The dimensional change results indicate that higher carbon alloys 1-3 have greater amounts of retained austenite and highly stressed plate or acicular martensite, due to the large growth resulting from the LNQ and the large shrinkage due to the tempering, respectively. Alloy 4 is relatively insensitive to post sintering thermal treatments. This is a result of the lower carbon content (0.53% sintered C) and no admixed copper. The improved hardenability of Alloy 4 via a combination of Cr, Ni, Mo and Si allows this lower carbon content to be used in sinter-hardening. The lower carbon content results in lath martensite formation, which is less stressed than the higher carbon acicular martensite. Shrinkage upon tempering is therefore reduced. The lower carbon and lack of copper greatly reduce the amount of retained austenite, thereby reducing the sintered compact growth upon LNQ. Alloy 4 had a much smaller growth (+0.06%) resulting from the LNQ quench as compared to Alloys 1-3 that had growths more than double this amount. Alloy 4 is therefore more dimensionally stable after sintering

Table 8. Hardness and TRS for all four alloys with the different thermal treatments.

Condition	Alloy 1 - FLC2-4808		Alloy 2 - FLNC-4408		Alloy 3 - FLC-4608		Alloy 4 - 4300	
	Hardness (HRA)	TRS (MPa)	Hardness (HRA)	TRS (MPa)	Hardness (HRA)	TRS (MPa)	Hardness (HRA)	TRS (MPa)
As-sintered	75	979	69	959	74	676	71	1690
LNQ	77	786	70	828	75	600	72	1593
T	71	1869	65	1517	69	1521	69	2062
LNQ + T	72	1621	68	1586	72	1362	70	1903
T + T	71	1807	66	1614	68	1528	69	2007

Hardness and strength of all four alloys studies is shown in Table 8. The as-sintered properties of Alloy 4 were the best of all alloys tested and the LNQ had little effect on properties. Tempering improved TRS and tensile properties by roughly 25%, and while significant for developing the best properties, tempering was much less important in this alloy system compared to Alloys 1-3. Again, this is a function of the martensite type that is present in the lower carbon steel. As-sintered hardness in Alloy 4 was lower than Alloys 1 and 3, but after tempering, Alloy 4 had similar hardness. The transverse rupture strength of Alloy 4 is equivalent or better than Alloy 1 (Table 8) and the tensile properties are similar. It should be noted that the sintered density of Alloy 4 was 0.05 to 0.10 **g/cm^3** higher than the other alloys due to a combination of improved compressibility and less growth during sintering. Given the ability to properly sinter a Cr-containing PM alloy, Alloy 4 provides a more dimensionally stable sinter-hardening system as compared with more traditional sinter- hardening alloys.

Summary:

We reviewed the benefit of using a 0.3 prealloyed Mo grade powder with FC-0208 type of powder both in as sintered and heat treated condition. Spur gears were produced and tested for dimensional control. For heat treated parts the use 0.3%Mo without Cu gives a better dimensional precision than FC-0208.

FC-0208 parts made with Ancorbonded process used in making higher density AncorMax 225 powder provides a tighter dimensional tolerance by ensuring all the graphite goes in to alloying compared a non- bonded mix.

In the case higher performance sinter-hardenable grades Ancorsteel 4300 provides good dimensional precision. Also shown are the benefits of tempering as a key part of maintaining dimensional precision in the case of sinterhardenable alloys .

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