

VACUUM SINTERING--A WAY TO IMPROVE DUCTILITY
AND TOUGHNESS OF STRUCTURAL P/M PARTS AT THE RIGHT PRICE

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ABSTRACT

Most parts fabricators do not understand the importance of a quality sinter to provide improved ductility and toughness for tomorrow's markets. As a result, the potential for growth markets for quality parts has seldom been exploited. Today the main emphasis is still on cost reduction, rather than quality.

It all starts with the powders. Even today's parts fabricators do not demand improved powders. Additives to powders are often deleterious to the properties of the parts, a fact of which most parts fabricators are unaware. This paper points out several factors that have not been previously explored and demonstrates how properties can be impaired through lack of attention to these minor additions. Comments will also be presented regarding selection of lubricants.

The paper will then present the advantages of vacuum sintering of FC0208 and FN0205 parts. Mechanical properties achieved through sintering at 1120°C (2050°F) or at 1260°C (2300°F) will be demonstrated. Emphasis will be placed upon vacuum sintering. The paper will compare these properties with those presently listed in Standard 35. The paper will then show the advantages of vacuum sintering for attaining toughness and quality of gear teeth.

Finally, a part will be selected for study. The part will be fabricated from a 0.45% phosphorus iron. Processing of the part will be described, including the sintering cycle provided. Tensile specimens will be included for quality control. The functional characteristics of the part will be described. A cost analysis will be presented to demonstrate that quality can be achieved at relatively low cost.

INTRODUCTION

Today's P/M parts are produced mainly in belt furnaces and sintered at temperatures of the order of 1120°C (2050°F). Most parts seldom experience more than 30 minutes above 1090°C (2000°F), many no more than 15 minutes. In addition, the length of time parts remain in the hot zone fixes the amount of time they experience during other important stages of the sintering process.

One such stage of processing includes burn-off of the lubricant. Ideally all lubricant should be eliminated cleanly from the part, leaving no evidence that the lubricant was ever present. Failure to eliminate lubricant from the part properly results in either carbon contamination or entrapment of gaseous lubricant, which will create large pores at the surface.^(1,2)

Although most lubricants liquify at approximately 250°C (500°F), the liquid remains entrapped within the part.⁽¹⁾ Not much happens until 430°C (800°F), the temperature at which most lubricants reach their vapor phase, is attained. At this temperature it now becomes easier to sweep the vaporized lubricant from the part, provided the part remains at this temperature for sufficient time to permit egress of the lubricant.

A belt speed that is set to permit parts to experience between 30 and 60 minutes above 1090°C (2000°F) to sinter the parts does not allow sufficient time below 540°C (1000°F) for complete removal of lubricant from the parts. Owing to the severe cold deformation experienced at the surface of the part, pores of high density parts (6.8-7.2 g/cm³) start to close at a temperature of 540°C (1000°F). This pore closure (sintering) prohibits transport of vaporized lubricant from the interior of the part to the surface where it can be eliminated.⁽²⁾

Owing to the importance of time allotted to sintering, cooling rates within belt furnaces are generally limited to the order of 50°C (120°F)/minute. For hardenable parts, this quench rate is often insufficient to permit transition of austenite to bainite or martensite.⁽³⁾

Other problems difficult to resolve with belt furnaces involve atmosphere control and ability to prevent leakage or seepage of the resident atmosphere into the furnace. Unless furnaces are tight and adequate atmosphere flow is achieved, contamination of the sintered parts is possible.

The use of a vacuum furnace can eliminate the problems that occur daily when parts are sintered in belt furnaces. The problems disappear because sintering in a vacuum furnace gives the engineer total control of the sintering process. Initially, to remove lubricant from the part, the engineer will create a vacuum. Once the parts are heated above 430°C (800°F), the lower external pressure in the chamber will permit the lubricant to be voided quickly, owing to the greater pressure inside the part. In addition, the furnace temperature can be maintained between 430°C (800°F) and 540°C (1000°F) until all the lubricant is voided.

Once the lubricant is removed, the furnace temperature can be elevated over a wide range, to sintering temperatures well beyond those achieved within belt furnaces, and maintained as long as desired, under any prescribed atmosphere. Once the parts are sintered, they can be control-cooled, such as in annealing, or they can be quenched to permit bainitic or martensitic microstructures. All this can be accomplished at reasonable cost savings.

The purpose of this paper is to demonstrate the versatility of vacuum sintering. The paper will describe how the process can be scaled up to sinter production parts. Examples of quality, derived from guaranteed properties, are shown, as well as a cost analysis to permit an understanding of the economics of vacuum sintering.

SINTERING OF RING GEARS OF STANDARD 35 MATERIAL COMPOSITION

Ring gears, as shown in Figure 1, were compacted from an FNO205 mix. Tensile and transverse rupture specimens were compacted from the FNO205 blend, and from an FCO208 blend as well. All specimens were compacted at a pressure of 680 MPa (50 TSI).

The ring gears, tensile, and transverse rupture, specimens were sintered at 1120°C (2050°F), and 1260°C (2300°F). Rings and specimens were loaded in a vacuum production furnace, Figure 2. The furnace was evacuated to less than 100 microns' pressure. The furnace temperature was then elevated to 540°C (1000°F) to remove the lubricant.

After removal of the lubricant from the ring gears, a partial pressure of hydrogen was introduced and the temperature was increased to 1120°C (2050°F), or 1260°C (2300°F), respectively. The ring gears were maintained at the respective sintering temperatures for one hour and then were permitted to furnace cool to 870°C (1600°F). The ring gears were finally pressure quenched in argon to ambient.

Density and dimensional change from die size of the sintered transverse rupture bars are shown in Table I. Note the densities and controllable dimensions that resulted from the imposed sintering conditions. The FCO208 size is especially of interest because, under these conditions, the parts exhibited zero growth.

The tensile properties are compared with those listed in Standard 35 in Table II.⁽⁴⁾ Note the similarity of the tensile properties of the FNO205 specimens. Since this is a nickel steel, and nickel does not promote bainitic or martensitic transformation in steel, but enters into solid solution, the tensile properties are not too different from those listed in Standard 35. However, as a result of the vacuum sinter, the nickel is more homogeneously absorbed in the microstructure, Figure 3. The FCO208 specimens sintered in the load present a different story, owing to the copper content of this alloy. Compared to those listed in Standard 35, the specimens sintered in the vacuum furnace exhibit vastly superior tensile properties.⁽⁵⁾ This material can be truly classified as an engineering material because it has higher strength, improved ductility and toughness, despite minimum hardenability contributed by the copper addition, owing to the improved sinter. Microstructure of the cross section of the FCO208 tensile specimen is shown in Figure 4.

In addition to tensile tests used to evaluate the sinter, teeth were sectioned from the gears and tested for impact strength using a specially modified Finius Olsen Model 74 Impact Tester capable of holding individual teeth. Production gear tests yielded average values of 17.86 ft. lbs. The 1120°C (2050°F) vacuum sintered gears yielded values similar to the production average (17.625 foot-pounds). However, the gears sintered at 1260°C (2300°F) yielded impact strengths of 24.5 ft. lbs., a 37% improvement.

PILOT RUN OF ELEMENT SUPPORT BUSHINGS FROM A 0.45% PHOSPHORUS IRON

We believe the cost of sintering an equivalent volume of parts per hour in a vacuum furnace is not greater than the cost of sintering in a belt, pusher, walking beam, or other furnace. In addition, vacuum sintering permits control of all stages of the sintering process, including burn-off of lubricant, sintering cycle, and cooling rate. Any protective atmosphere, including hydrogen or absence of atmosphere (vacuo) can also be selected, or changed, at any stage of processing, for the benefit of producing quality product.

To demonstrate both technical and economical viability, an element support bushing, Figure 5, was selected for evaluation. The goal of the program was to sinter effectively 1,000 parts in a vacuum production furnace, as shown in Figure 6.

Prior to sintering the 1,000 element support bushings, a dummy run consisting of tensiles, transverse rupture specimens and rings of 0.45% phosphorus iron, was loaded into the furnace, as shown in Figure 7, to demonstrate uniformity of properties. Specimens located at sites 7 and 8 were elevated on steel bars to simulate tiers of load as in a production mass. Five tensile specimens, five rings, and three transverse rupture specimens were located at each of the eight individual sites. Three work thermocouples were distributed, as shown, to demonstrate temperature uniformity. After loading, the furnace was evacuated to a pressure of 100 microns, and the temperature was raised to 540°C (1000°F) to permit removal of lubricant under a partial pressure of vacuum. Pure hydrogen was then introduced and the temperature was increased to 1120°C (2050°F), and maintained for one hour, after which the specimens were cooled in the partial pressure of hydrogen to ambient.

Green properties of the specimens are shown in Table III. These properties are typical of a 0.45% phosphorus iron compacted at 680 MPa (50 TSI). The tensile properties that resulted from the sintering process are shown in Table IV. These properties are compared with the properties achieved in a laboratory furnace employing an equivalent sintering cycle. It is obvious from the data that properties did not vary appreciably regardless of where the specimens were positioned in the furnace. None of the specimens exhibited less than 14% elongation and all had a yield strength greater than 230 N/mm² (34,000 psi). The specimens sintered to densities greater than 7.4 g/cm³, and all exhibited uniform shrinkage of less than 0.3% from die size. The experiment clearly demonstrated that parts with uniform properties of exceptional quality could be manufactured in a production furnace as well as in a laboratory furnace.

Magnetic properties and interstitial analyses are shown in Table V. These data are included only to show further the uniformity of the properties since the requirements of the element support bushings were mechanical rather than magnetic. Analyses of carbon, nitrogen, oxygen and sulphur also demonstrate the purity of the specimens and the uniformity of control regardless of position within the furnace.

PRODUCTION RUN OF ELEMENT SUPPORT BUSHINGS

Armed with the knowledge of property uniformity, element support bushings (Figure 5) were compacted from a 0.45% phosphorus iron. Two separate runs were made using the cycle described previously. One run consisted of 750 element support bushings, the second of 1,000 element support bushings. Since one picture is better than a thousand words, the load of 1,000 element support bushings prior to insertion into the furnace is shown in Figure 8. Tensile and transverse rupture specimens were located as shown in Figure 9. The sintering cycle employed was similar to that used in the pilot study. Green and sintered density, and dimensional changes, are shown in Table VI. These data are similar to that experienced previously.

A summary of the tensile properties and interstitial contaminants present after sintering is shown in Table VII. Again, most of the elongations were 15% or greater, with a standard deviation of 4%, and all yield strengths were greater than 240 N/mm² (35,000 psi), with a standard deviation of 5 N/mm² (700 psi). Only specimens located at the rear of the bottom tier had elongation less than 15%.

When the data is compared with that in the pilot run (Table IV), it is clearly seen that the process can be scaled up to production loads that produce parts of guaranteed superior quality.

This part was originally machined from AISI 1010 bar stock. By incorporating some additional features, sufficient savings in machining costs resulted in a savings of 2/3 that of the original cost. The part is now in service and no complaints regarding its performance have been received. In fact, praise has been the byword.

THE FUTURE AND BEYOND

A product has been manufactured here which suggests a way to expand the P/M market to produce parts not envisioned today. The parts described exhibit uniformity in production, lot to lot, consistent yield strength, 15% or more elongation, and improved toughness, all at lower cost. Parts such as these made using P/M technology should be of interest in the future.

However, as with any new development, problems have emerged with this process. Burn-off of lubricant is not as clean as desired. When production quantities are sintered, some lubricant remains within the furnace. Since production of quality, clean, parts is desired, a contaminated furnace is not appreciated. We are working on an improved method to burn-off lubricant in a production furnace so that no residue remains. We believe we have a solution, however it is too early to report this information. Perhaps this will be substance for a paper next year.

Another concern is fixturing of P/M parts when loading the vacuum furnace. The parts must be fixtured to prevent them from sintering together and also from fusing with the supports on which they rest. More work is necessary to establish material for reusable, inexpensive supports that will not contaminate the parts, while providing adequate support at today's, or even higher, sintering temperatures.

The promise of improved parts of consistent quality at reasonable cost has been demonstrated. Now the bugs which plague all new processes must be solved to maximize quality and profits.

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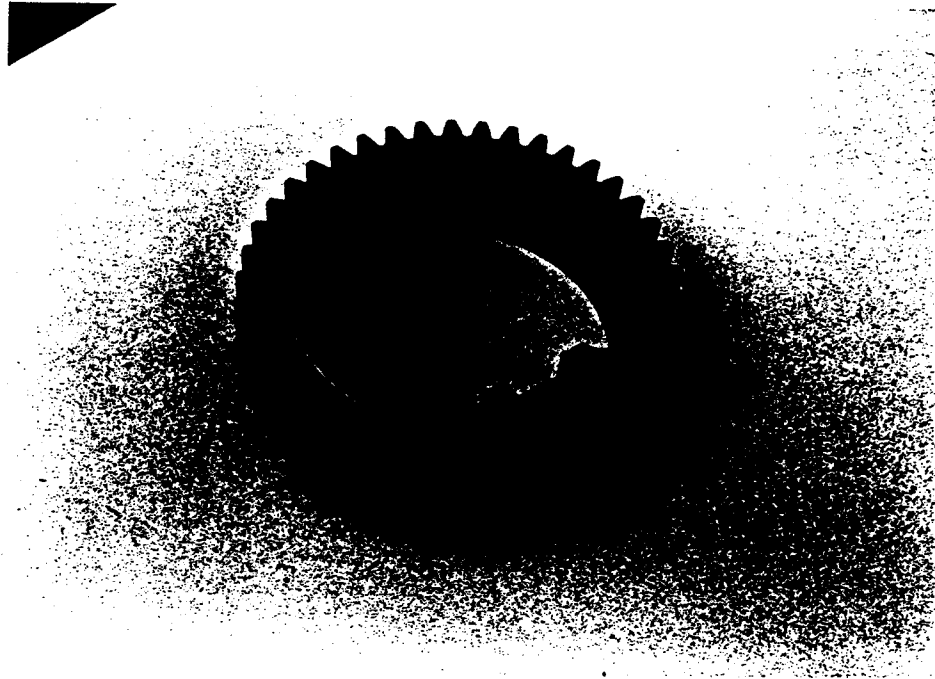


Figure 1. Ring Gear

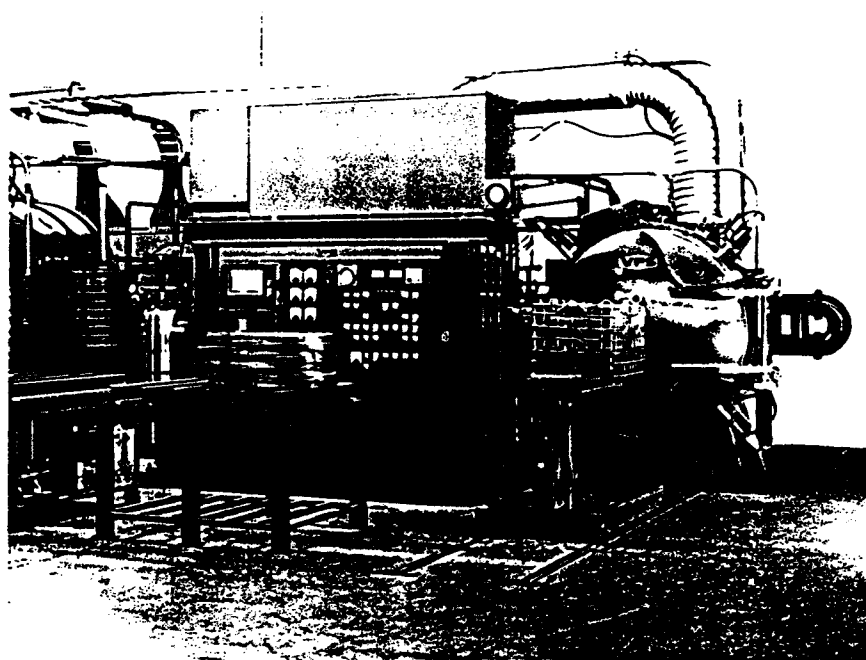


Figure 2. Production Furnace

Table I

Green and Sintered Characteristics of Specimens
Sintered In Vacuum Production Furnace With Sinteris Gears

<u>Property</u>	<u>FN0205</u>		<u>FC0208</u>
	<u>2050°F</u>	<u>2300°F</u>	<u>2300°F</u>
Green Density - g/cm ³	7.09	7.09	7.07
Green Expansion From Die Size - %	0.45	0.45	0.35
Stripping Load - Pounds	1800	1800	1600
Sintered Density - g/cm ³	6.99	7.23	7.16
Sintered Change in Length from Die Size - %	0.48	-0.16	-0.006

Table II

Tensile Properties of Specimens
Sintered In Vacuum Production Furnace With Sinteris Gears

<u>Property</u>	<u>FN0205</u>	<u>FN0205 (0.4C)</u>		<u>FC0208</u>	<u>FC0208 (0.68C)</u>
	<u>Std. 35</u>	<u>2050°F</u>	<u>2300°F</u>	<u>Std. 35</u>	<u>2300°F</u>
Ultimate Tensile Strength - psi	70,000	63,100	71,800	75,000	103,600
Yield Strength (0.2% Offset) - psi	40,000	36,300	44,400	65,000	83,600
Elongation - % in 1 Inch	5.5	4.3	6.4	<1.0	3.2
Apparent Hardness - HRB	78	72	72	84	89

Chemical Analysis

<u>Element</u>	<u>FN0205</u>		<u>FC0208</u>	<u>Gear</u>	<u>Gear</u>
	<u>2050°F</u>	<u>2300°F</u>	<u>2300°F</u>	<u>2050°F</u>	<u>2300°F</u>
C	0.41	0.42	0.68	0.41	0.42
N	0.0006	0.0002	0.0000	0.0003	0.0004
O	0.0289	0.0281	0.0305	0.0353	0.0529
S	0.0112	0.0103	0.0073	0.0043	0.0074

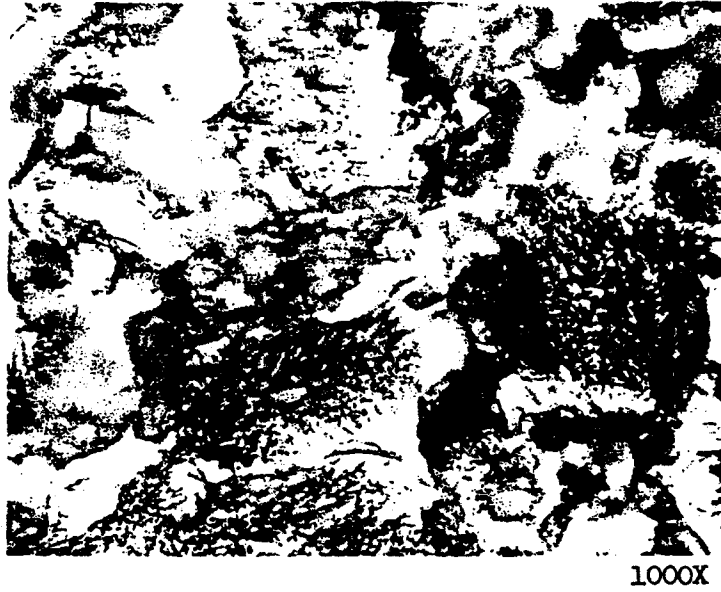
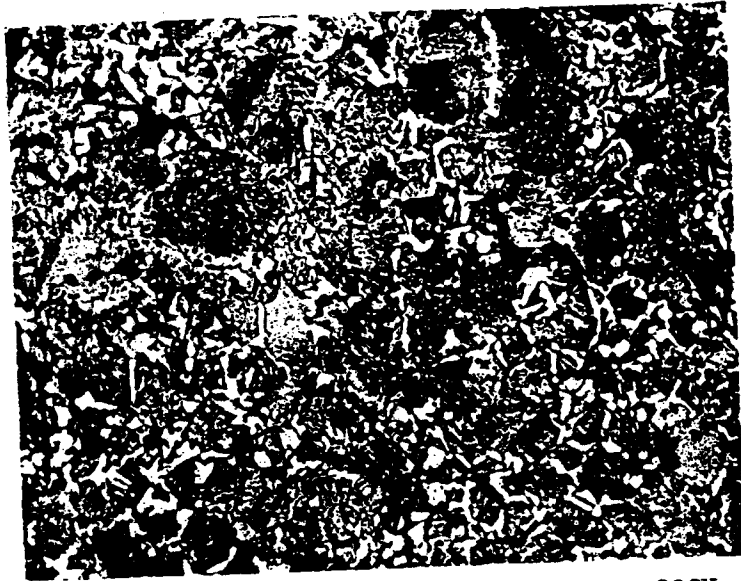


Figure 3a. Microstructure of Ring Gear Sintered at 1120°C (2050°F).



Figure 3b. Microstructure of Specimen Sintered at 1260°C (2300°F).



200X

Figure 3c. Microstructure of Gear Sintered at 1260°C (2300°F).



1000X

Figure 4. Microstructure of FC-0208 Specimen Sintered at 1260°C (2300°F).

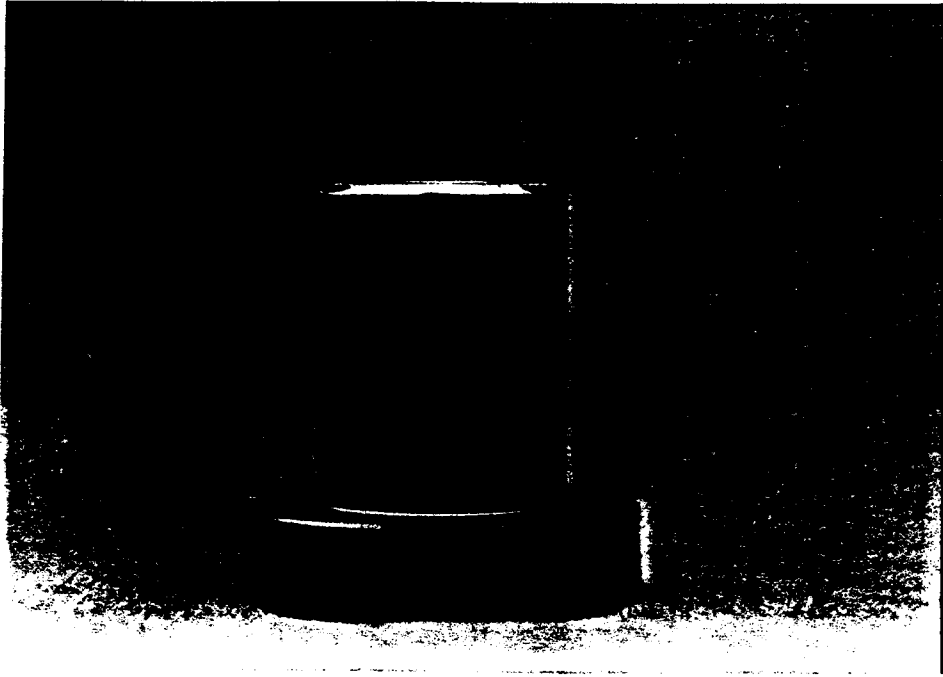


Figure 5. Support Bushing

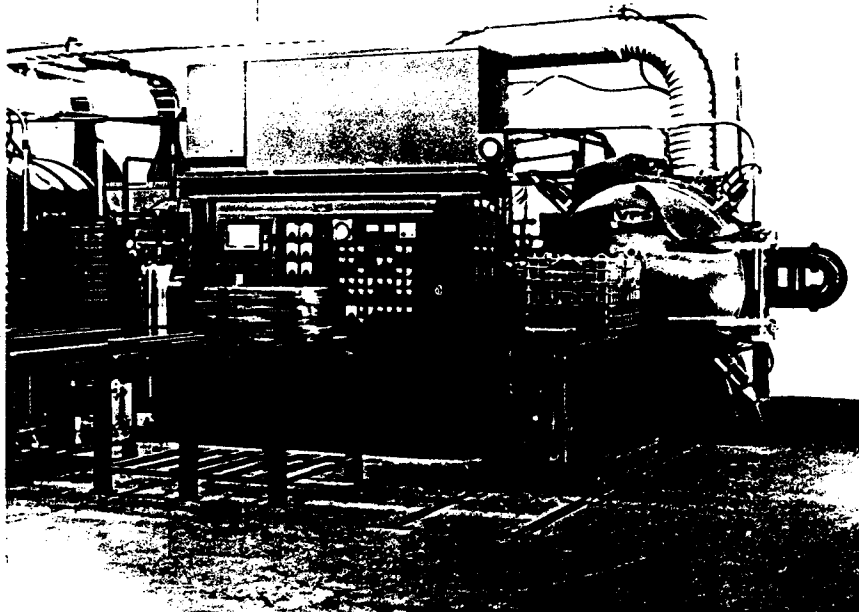


Figure 6. Production Vacuum Furnace

Furnace HT-10

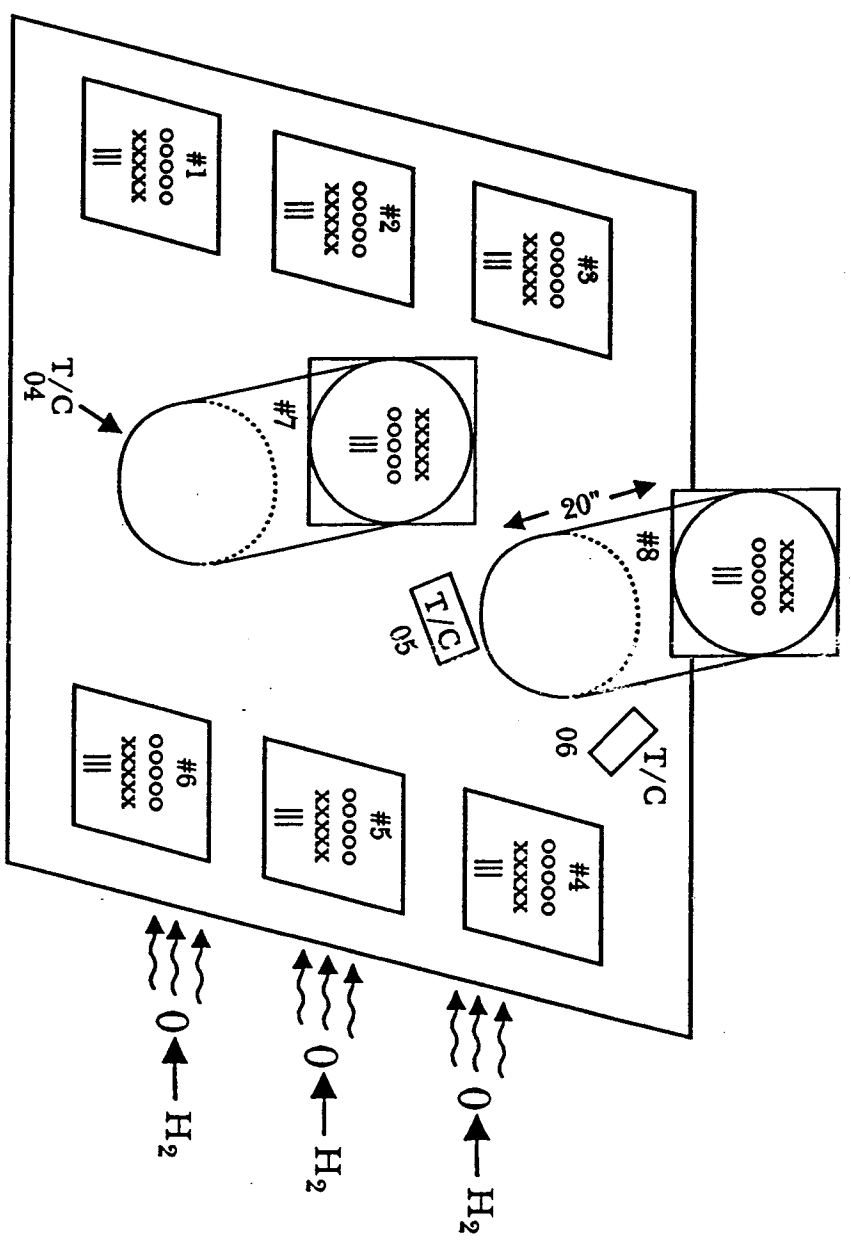


Figure 7. Location of Sets of Test Specimens

Table II I

Green Properties of Phosphalloy
45 Specimens Sintered in Furnace 10

<u>Site</u>	<u>Green Density - g/cm³</u>	<u>Expansion From Die Size - %</u>
1	7.17	0.35
2	7.15	0.36
3	7.16	0.35
4	7.13	0.35
5	7.14	0.37
6	7.14	0.35
7	7.15	0.38
8	7.14	0.37
Lab	7.15	0.36

Table IV

Tensile Properties Of Phosphalloy 45 Specimens Sintered In Furnace 10

Property	Site 1	Site 2	Site 3	Site 4	Site 5	Site 6	Site 7	Site 8	Lab
Ultimate Tensile Strength - psi	54,700	52,900	53,600	53,900	54,300	53,900	53,100	54,200	55,700
Yield Strength (0.2% Offset) - psi	35,800	34,600	35,800	34,100	35,000	34,600	34,500	34,900	36,400
Elongation - % in 1 inch	15.0	14.0	14.0	14.0	15.0	15.0	15.0	15.0	16.0
Apparent Hardness - HRB	58	61	60	61	60	60	61	59	62
Sintered Density - g/cm ³	7.43	7.46	7.43	7.45	7.45	7.45	7.46	7.44	7.43
Shrinkage from Die Size - %	0.30	0.27	0.27	0.28	0.26	0.27	0.32	0.25	0.16

Table V

Magnetic Properties Of Phosphalloy 45 Rings Sintered In Furnace 10

<u>Property</u>	<u>Site 1</u>	<u>Site 2</u>	<u>Site 3</u>	<u>Site 4</u>	<u>Site 5</u>	<u>Site 6</u>	<u>Site 7</u>	<u>Site 8</u>	<u>Lab</u>
Magnetic Induction At An Applied Field of 15 Oe	13,300	13,600	13,600	13,700	13,700	13,600	13,600	13,600	13,500
Remanent Magnetization - Gauss	12,800	12,900	12,800	13,000	13,000	13,000	13,000	12,900	12,800
Relative Maximum Permeability	5,900	6,000	6,000	6,200	5,800	6,000	5,700	5,800	5,500
Coercive Force - oersteds	1.2	1.2	1.2	1.2	1.2	1.2	1.3	1.2	1.2
Sintered Density - g/cm ³	7.43	7.46	7.43	7.45	7.45	7.45	7.46	7.44	7.43
Average Grain Size - Microns	68	65	60	60	62	63	57	64	66

Chemical Analysis - %

C	0.0006	0.0012	0.0010	0.0007	0.0008	0.0001	0.0030	0.0002	0.0020
N	0.0005	0.0004	0.0004	0.0004	0.0004	0.0003	0.0004	0.0006	0.0012
O	0.0512	0.0490	0.0507	0.0504	0.0506	0.0575	0.0543	0.0558	0.0354
S	0.0030	0.0029	0.0031	0.0028	0.0027	0.0029	0.0034	0.0029	0.0030

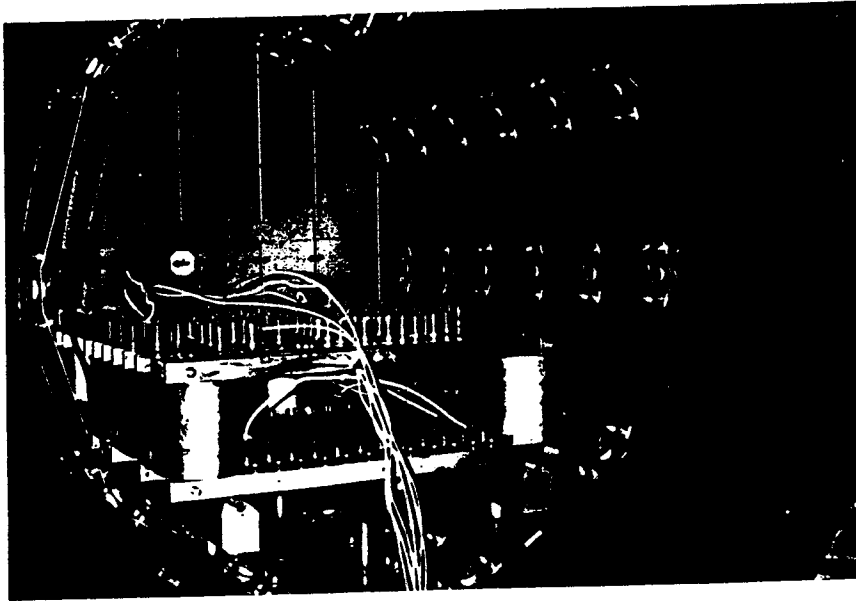
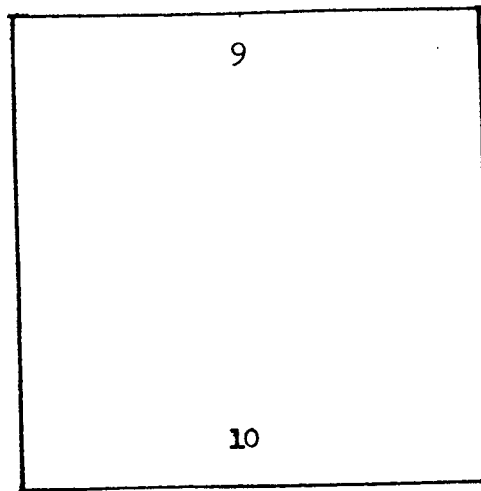
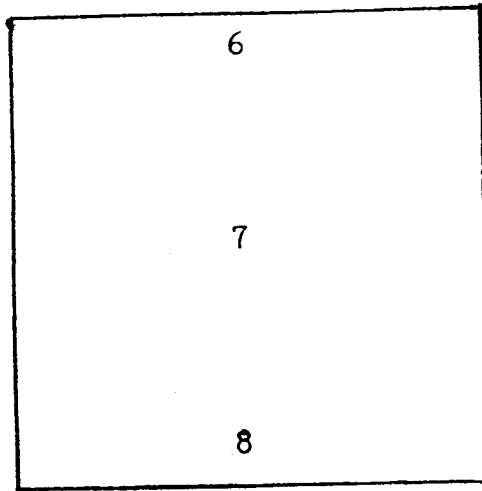


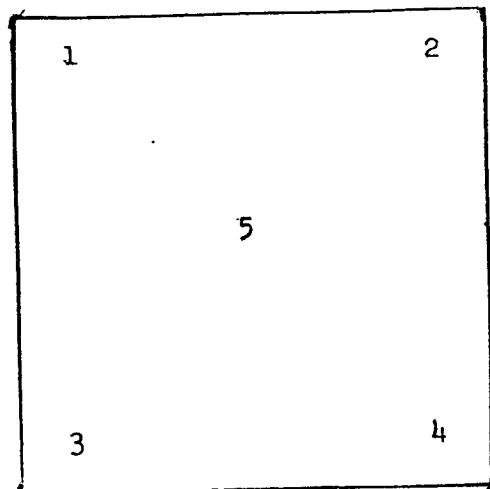
Figure 8. Arrangement of Lot as Prepared for Sintering



Top Tier



Middle Tier



Bottom Tier

Figure 9. Arrangement of Control Specimens for 1000 Piece Sintering Cycle

Table VI

Mechanical Properties of Specimens
Sintered With 1000 Element Support Bushings

<u>Property</u>	<u>Green Properties</u>			
	<u>TRS Specimen</u>	<u>Whole</u>	<u>Part Flange</u>	<u>Shank</u>
Stripping Pressure - lbs.	2000	-----	-----	-----
Green Density - g/cm ³	7.13	6.56	6.90	6.40
Expansion from Die Size - %	0.38	-----	-----	-----
<u>Sintered Properties</u>				
Sintered Density - g/cm ³	-----	6.77	6.86	6.42
Shrinkage from Die Size - %	-0.26	-----	-----	-----

Table VII

Tensile Properties of Specimens Distributed In 1000 Piece Run

<u>Specimen</u>	<u>Ultimate Tensile Strength - psi</u>	<u>Yield Strength (0.2% Offset - psi)</u>	<u>Elongation % in 1 inch</u>	<u>Apparent Hardness-HRB</u>
1	55,200	37,000	14	61
2	55,100	35,600	12	61
3	55,300	35,100	16	63
4	55,700	35,300	19	64
5	59,100	36,400	23	60
6	57,500	36,800	18	61
7	57,500	36,700	18	61
8	56,600	35,900	20	61
9	55,200	35,300	24	62
10	58,200	36,600	20	60
Mean	56,400	36,100	18	61
Standard Deviation	1,600	700	4	5

Chemical Analysis

<u>Element</u>	<u>Tensile Specimen</u>	<u>Support Bushing</u>
C	0.003	0.005
N	0.0255	0.0489
O	0.0403	0.0415
S	0.005	0.005