

PROCESSING EXPERIENCE OF GREEN STRENGTH ENHANCED MATERIAL SYSTEMS

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ABSTRACT

Green strength enhanced material systems have been developed for iron and Low alloy as well as stainless powder metallurgy applications. Relative to normal processing, the increase in green strength is 50-100%. The nature of green strength with respect to both materials and processing conditions is reviewed. The processing variations designed to meet target properties such as apparent density, flow and compressibility are compared with conventional material systems. Manufacturing experience with a mechanical press is presented.

INTRODUCTION

Green strength is playing an increasing role in powder metallurgy (P/M) processing due to the increased demand for robust green compacts. As the P/M processing line becomes more automated and dimensional tolerance becomes more critical, the need to enhance green strength and reduce green scrap is critical. Sufficiently high green strength permits green machining [1], an attractive alternative to machining sintered parts to desired dimensional tolerances. Recent advances in enhanced premixes [2,3] have resulted in the development of the ANCORMIX material system. The first commercial product in the ANCORMIX family contains Ancor GS-6000, a lubricant system designed to enhance the green strength of green compacts made with stainless steel, iron and Low alloy powders.

This is the second paper to address the processing of materials that contain Ancor GS-6000. In the first paper [3], the Ancor GS-6000 lubricant system is referred to as S-lube. Variables examined during the present study include:

- Effect of pressing temperature during production compaction
- Effect of lithium stearate additions to the Ancor GS-6000 (dilution) - effect of lubricant particle size and the resultant pore morphology - production variation with Ancor GS-6000 additions

The relationship between green strength, lubricant content and compaction pressure in green compacts has been investigated by other researchers. [4,5,6] Typically, unlubricated powder had significantly higher green strength than a powder with internal lubricant. [7] For a specific powder, green strength can be affected by the geometric and topographical factors of the powder mixture as well as surface related factors. All these factors are influenced by the temperature during compacting. Using the ANCORDENSE[®] process, compaction at temperatures in the range of 265°F to 310°F (130°C to 155°C) increases the green and sintered properties of P/M parts. [8] The study of material properties at various temperatures has been reported previously. [9] However, little work has been reported of the influence of temperature in the range of 75°F to 215°F (24°C to 102°C) on the processing of green compacts.

Since most PIM lubricants are solid phases with melting points above 102°C, the effect of temperature within this range typically is assumed to be negligible. While the effect of lubricant particle size on P/M processing has been reported [10], the effect of a softening lubricant on green strength and green expansion has not been reviewed. Knowledge of green expansion and green strength enhancement allows the P/M part fabricator to design the compact properly and to reduce the likelihood of green cracks. [11,12]

Typically, higher green densities lead to greater green expansion. For a complex-shaped part, there can be large density variations between different sections of the compact. During ejection, one section may expand at 3 to 4 times that of a neighboring section, causing the compact to form cracks or laminate at the section boundary. Green expansion is a measurement of the dimensional spring back of the compact in three dimensions after it is ejected from the die cavity. Typically, the spring back relative to the die is considered in P/M processing and is defined as:

$$\text{Green Expansion (\%)} = 100\% \times (L - D)/D$$

Where

L = size of the green compact

D = size of the die cavity

This change in the dimension of the part is due to the release of stress stored in the compact. This results in a green compact that is larger than the dimension of the die cavity. A typical value for green expansion is less than 0.3%.

EXPERIMENTAL PROCEDURE

Laboratory testing procedures were carried out in accordance with appropriate ASTM standards. Table I lists the laboratory premixes used to examine the effects of pressing temperatures, dilution effects and particle size of Ancor GS-6000 additions. To evaluate the properties in the green and sintered states, transverse rupture strength (TRS) bars were prepared according to ASTM B 312. The reported values are the average of three bars. The TRS bars from the 410L stainless steel lubricant combination study and the Ancor GS-6000 particle size study were pressed at 145°F to simulate the production environment and to standardize the pressing temperature.

Ancor GS-6000 is a patented lubricant system developed by Hoeganaes. All other ingredients used in this study are available commercially. The lithium stearate was produced by Witco, Acrawax-C was from Lonza, 8081 copper from Alcan, and the 3203HS graphite was from Asbury.

In the compaction temperature study on 303L with 2.25 w/o copper and 1 w/o lubricant, the pressing temperatures evaluated were 75°F (room temperature), 110°F, 145°F, 180°F and 215°F (43°C, 63°C, 82°C and 102°C, respectively). The compaction pressures tested at those temperatures were 30 tsi and 50 tsi (414 MPa and 690 MPa), respectively.

For the stainless steel premixes, sintering conditions were 2350°F (1288°C) in a hydrogen atmosphere for 30 minutes at temperature. For the FN-0205 premix, sintering conditions were 2050°F (1121 °C) in 75 v/o hydrogen/25 v/o nitrogen atmosphere for 30 minutes at temperature.

The work is presented in the following sequence:

- Effect of pressing temperature on green properties of Ancor 303L stainless steel materials
- Effect of lithium stearate additions to Ancor GS-6000 (dilution) on Ancor 410L stainless steel materials
- Effect of lubricant particle size and resultant pore morphology on Ancor 410L and FN-0205 materials
- Production experience with Ancor 410L stainless steel materials

RESULTS AND DISCUSSION

The Effect of Pressing Temperature on Green properties of 303L Stainless Steel Materials

Initially, the green strength of an Ancor 303L stainless steel premix with 2.25 w/o copper and 1.00 w/o lubricant was evaluated with respect to temperature. During the compaction process heat is generated, initially, by inter-particle friction followed by die-wall to compact friction during ejection. Although heat is dissipated through the die table and the press frame, heat energy is stored in the compact during and after ejection from the die. Measurement of surface temperature on the ejected compact and the die surfaces indicates a substantial temperature increase. Depending on the powder chemistry, type of lubricant, part geometry, tooling design, pressing rate and pressing tonnage, this temperature increase can vary from a few degrees to over 215°F (102°C). Over time, thermal equilibrium is attained and the compaction process reaches a steady operating temperature. The stability of this temperature depends on the quality of the powder lubricant and the pressing variables. Typically, the taller the part, the higher the operating temperature. As the temperature increases, all the green properties of the compact will change.

In this study, the laboratory die and the punches were heated to uniform temperatures of 75°F, 110°F, 145°F and 180°F (24°C, 43°C, 82°C and 102°C, respectively). The powder was transferred to the die cavity and pressed into standard TRS bars, 0.5 inches in height. At these operating temperatures, the carbide die acts as a massive heat sink. Reproducibility of the

results was checked through multiple runs for a given compacting pressure and temperature. The variation between runs was less than 1%. This procedure simulates the production situation where cold powder is filled into a warm die. The effect of temperature on green properties is tabulated in Table II.

The effect of temperature on green strength at a compacting pressure of 50 tsi is shown in Figure 1. Material IC (lithium stearate) shows an increase in green strength from 990 psi (6.8 MPa) to 2350 psi (16.2 MPa) as the temperature increases from 75°F to 215°F. For a temperature increase of 140°F, the green strength is doubled. The rate of increase at 10 psi/°F indicates a pronounced linear relationship between green strength and compaction temperature. The increase for Material IC compacted at 30 tsi is shown in Figure 2. The green strength increases from 440 psi (3.0 MPa) to 1300 psi (9.0 MPa) as temperature increases from 75°F to 145°F (24°C - 102°C), with a corresponding 6-psi/°F rate of increase. This is understandable as the melting point of lithium stearate is 428°F - 430°F (220°C - 221°C). There is no softening or phase change as the operating temperature increases from 75°F to 215°F. The rate of increase in the green strength can be attributed to the reduction of yield stress in the compact. The movement of material during the compaction stage is an anisotropic deformation process. On an atomic scale, this is accomplished by either dislocation movement or diffusion. At such a Low temperature, the diffusion coefficients are correspondingly small. As the temperature increases, the effect of strain hardening diminishes, resulting in higher density for most powder systems and compacting pressures.

For Materials IA (Ancor GS-6000) and IB (50/50 ratio of Ancor GS-6000 and lithium stearate) green strength is linearly proportional to the temperature up to 145°F as shown in Figure 1. At a compaction pressure of 50 tsi, the green strength increases from 3400 psi to 4700 psi for IA. This is an increase of 1300 psi (9.0 MPa) for a temperature increase of 70°F. The rate of increase is 19 psi/°F which is twice that for Material IC. At 180°F, there is a significant increase in green strength to 7000 psi (48.3 MPa). A further increase in temperature to 215°F results in a slight reduction of green strength to 6800 psi (46.9 MPa). This is due to the fact that at 180°F, the Ancor GS-6000 lubricant softens and moves into the pore spaces. This increases powder movement and reduces residual stresses. The end result is enhanced particle-to-particle interlocking. Raising the temperature above the softening point to 215°F will not increase the green strength further. It is noted that the increase in green strength at 180°F is less pronounced at compaction pressures of 30 tsi (Figure 2) compared with 50 tsi. This indicates that the optimum benefits are achieved by a combination of both temperature and pressure.

As shown in Figure 1, the increase in green strength of Material IB followed the trend of Material IC up to 110°F. Increasing the temperature to 145°F and above, the trend of IB more closely represents that of the Ancor GS-6000 mix. The green strength achieved at 180°F is 3000 psi (20.7 MPa). This is an increase of 50% over the lithium stearate mix. The advantage of mixing lithium stearate to Ancor GS-6000 lubricant is to regain the density lost at lower compaction temperatures. As shown in Figure 3, the density achieved at 180 F for Material IA is almost equivalent to that of IC.

The effect of temperature on green density is shown in Figures 3 and 4. At a compaction pressure of 50 tsi, Material IC shows an increase in density from 6.65 g/cm³ to 6.85 g/cm³; an increase of 0.2 g/cm³ for a temperature increase of 140°F (60°C). At a compaction pressure of 30 tsi, the increase is reduced to 0.15 g/cm³. This is due to different packing and deformation patterns resulting from the two different compaction pressures. [3] Green density also increases with increasing temperature for Material IA. The rate of increase is higher for IA than for IC. At a compaction pressure of 50 tsi, IA increases in density from 6.39 g/cm³ to 6.82 g/cm³ as temperature increases from 75°F to 215°F (24°C to 102°C).

The effect of temperature on green expansion with respect to compacting pressure is shown in Figure 5. For a given compaction pressure, as temperature increases the green expansion decreases. There is a significant decrease in green expansion at 180°F (82°C) and above for all three lubricant mixes, The fact that Ancor GS-6000 softened at 180°F has little impact on the value of green expansion in this dimension. This is due to the corresponding residual stress relief within the die cavity. The residual stress is released first through the top and bottom direction as the top punch retracts from the compact. At this point the green compact will spring back (expand) in the pressing direction. The majority of the remaining residual stress is radial stress, corresponding to the energy stored in the metal to metal particle contact points. The stress stored in the lubricant is minimal.

Temperature has the most influence on the relief of radial stress. For a compaction pressure of 30 tsi, the green expansion can decrease from 0.1 to 0.01% as temperature increases from 75°F to 215°F. This indicates that temperature can be used as a means to reduce green expansion and related internal stress concentrations within the compact. In general, green expansion is inversely proportional to temperature. A better understanding of the green expansion characteristics would provide fabricators with information about the relationship between green cracks and excessive expansion of the compact.

The effect of temperature on ejection is shown in Figures 6 and 7. Ejection performance is measured as stripping pressure and sliding pressure. Stripping pressure is defined as the minimum force per unit area required overcoming the static friction between the green compact and the die wall. Sliding pressure is defined as the force per unit area required moving the green compact from the stationary position to the upper edge of the die cavity. The results indicate that there is a significant increase in stripping pressure at 180°F for Materials IA and IB. The increase can be as high as 500 psi relative to Material IC. At 180°F, the lubricant level at the die surface is actually reduced. This is due to relief of residual stress within the die cavity as discussed earlier. When the green compact expands in the height direction, the softened lubricant is pulled away from the die surface. Residual stress is finally released as the compact is ejected out of the die cavity. However, this is not the case for Material IC where the lubricant does not move and the lubricant level at the die wall remains constant. The effect on sliding pressure is shown Figure 7. A significant decrease in sliding pressure is observed at temperatures of 110°F and above. Overall, the increase in ejection force for the Ancor GS-6000 mix is relatively small and will not impact most compaction processes even at 180°F and above.

Using Ancor 410L stainless steel powder as base material for the laboratory premixes, 1 w/o total lubricant was admixed to the base material. Combinations of Ancor GS-6000 and lithium stearate were used to study the effect of minimizing property differences other than green strength. While the total lubricant level in the material was kept constant, the lithium stearate and Ancor GS-6000 contents were varied from 0 to 1.0 w/o in increments of 0.25 w/o. The dilution of the Ancor GS-6000 with lithium stearate in the Ancor 410L stainless-steel resulted in a change among most of the green and sintered properties. The data are shown in Tables III and IV. The strip and slide pressure results do not show a noticeable change with varying ratio of lubricants. Green expansion in the direction normal to the die wall does not show any dependency on the Ancor GS-6000 concentrations. To produce the TRS bars for this lubricant combination study, the die was heated to 145°F and then the bars were compacted at a designated pressure.

Apparent density and Hall Flow are inversely proportional to the concentration of Ancor GS-6000 in the premix. The apparent density of Material IIA is 0.39 g/cm³ lower than that of Material IIE. Material IIB has an apparent density close to IIE; while still increasing green strength more than 2.5 times that of Material IIE, at 30 and 50 tsi compaction pressures. Material IIA with 1 w/o Ancor GS-6000 exhibits a flow that is 16 seconds faster than Material IIE which contained 1 w/o lithium stearate. Incremental additions of Ancor GS-6000 to the lithium stearate improve the flowability.

At the compaction temperature of 145°F, the green density increases with increasing lithium stearate additions. However, the green strength decreases substantially. As discussed above, Material IIB optimizes the die fill properties, while still providing exceptional green strength levels.

For Material IIB, the green density is greater by 0.11 g/cm³ at 30 tsi and 0.06 g/cm³ at 50 tsi relative to Material IIA. The green density of Material IIE increases by 0.15 g/cm³ at 30 tsi and 0.07 g/cm³ at 50 tsi relative to Material IIA. After sintering, the density difference between materials increases further.

Table IV indicates a sintered density difference of 0.28 g/cm³ at 30 tsi and 0.15 g/cm³ at 50 tsi between Materials IIA and IIE, respectively. This difference clearly affects the transverse rupture strength (TRS) and hardness values. Figure 8 shows TRS increasing as the percentage of lithium stearate increases. Figure 9 shows a similar trend for hardness.

Figure 10 shows the TRS plotted as a function of sintered density for Materials IIA, IIB, IIC, IID, and IIE, at compaction pressures of 30 and 50 tsi. The resulting trend indicates that TRS is a direct function of sintered density. Figure 11 shows a similar trend for hardness. In order to achieve parity in sintered properties, the results indicate the need to minimize the differences in density between the Ancor GS-6000 and the lithium stearate systems. As previously discussed, green density is a function of compaction temperature. Increasing the compaction temperature above 145°F for the Ancor GS-6000 system would increase both green and sintered density. Further improvements in sintered density could also be obtained by increasing sintering time at temperature.

Ancor GS-6000 Particle Size Effects on 410L Stainless Steel Materials

As the study on lubricant combinations points out, there is degradation in sintered properties due to lower density. Examination of the pore structure in the sintered TRS bars indicated that the pores of Material IIA (Figure 12) are larger and occupy a greater cross-sectional area than Material IIE (Figure 13). These observations led to the study on the effects of Ancor GS-6000 particle size on sintered mechanical properties. The relationship between the porosity and the sintered

properties required further investigation to determine if larger pores contribute to the decrease in sintered properties.

Material IIF was produced using a finer Ancor GS-6000 addition (finer than 20 μ m). Property data from the regular Ancor GS 6000 (IIA), lithium stearate (IIE), and the 50/50 ratio combination of Ancor GS-6000 and lithium stearate (IIC), are compared with Material IIF in Table V. At 30 tsi compaction pressure, the resultant sintered density of Material IIF increased by 0.04 g/cm³ compared with Material IIA but the TRS and hardness values did not improve. At 50 tsi, the resultant sintered density was near parity between Materials IIA and IIF and the sintered strength increased by 12,000 psi (82.8 MPa), or 10% for Material IIF.

The finer particle size lubricant in Material IIF results in a more rounded, smaller, and uniformly distributed pore structure as compared with the regular Ancor GS-6000 in Material IIA. This can be seen by comparing the photomicrographs shown in Figures 13 and 16. The pore structure of Material IIF looks similar to that of Material IIC (50/50 mixture of Ancor GS-6000 and lithium stearate) as seen in Figures 15 and 16. Materials IIC and IIF had a higher frequency of irregular pores compared with IIE. The pores resulting from using a coarser Ancor GS-6000 addition are shown in Figure 16. The increased frequency and size of irregular pores is related to the particle size of the Ancor GS-6000.

Ancor GS-6000 Particle Size Effects on FN-0205 Materials

To study the effects of Ancor GS-6000 particle size on conventional P/M materials, the performance of a FN-0205 premix was evaluated. The regular and fine particle size Ancor GS-6000 additions were compared with a 1.0 w/o Acrawax addition. Table I lists the material compositions. TRS bars were pressed at 30, 40, and 50 tsi compaction pressures and at a compaction temperature of 145°F. The results are tabulated in Table VI. The green strength for both materials with Ancor GS-6000 (IIIA and IIIB) is greater than 2.5 times that of the premix with Acrawax (IIIC). The fine particle material (IIIA) provided the maximum green strength (5,500 psi), while minimizing the apparent density difference with respect to Material IIIC.

Sintered property results are listed in Table VII. All materials were sintered under conventional conditions; 2050°F (1121°C), in a 75 v/o hydrogen/0.25 v/o nitrogen atmosphere. The finer Ancor GS-6000 (IIIA) provides sintered density, TRS and hardness near parity to Material IIIC, at 30 and 40 tsi.

Figure 17 shows the TRS of the three materials and at 50 tsi, the TRS of Materials IIIA and IIIB are lower than that of Material IIIC. This disparity in TRS is commonly associated with laminations due to highly compressible ingredients. Figure 18 shows that the finer particle size Ancor GS-6000 improves the apparent hardness; as would be expected with the sintered density increase.

Production Experience with 410L Stainless Steel Materials

An Ancor 410L stainless steel powder premix with Ancor GS-6000 was compared with a conventional premix of Ancor 410L with lithium stearate, compacted under production conditions. Each mix contained 0.75 w/o lubricant addition. Prior part production Using lithium stearate posed green strength and handling difficulties for the customer. An illustration of the part is presented in Figure 19. The thin wall (0.03125 inches (0.79375 mm)), and large height to diameter ratio (4), often resulted in part inconsistencies; In addition, high green scrap levels were experienced due to Low part green strength.

Production trials were conducted on a single action, 60-ton compaction press. Part chipping and occasional part disintegration during production was observed with the lithium stearate premix. These problems were immediately eliminated when testing the Ancor GS-6000 lubricant system. Overall length (O.A.L.) and part mass data were collected according to the fabricator's normal quality control procedures; Ten parts were sampled every 30 minutes of production (approximately 750 parts per hour of production). The surface temperature of the compacts varied from 110°F (43°C) to 120°F (50°C).

The average O.A.L. and part mass were recorded for continuous production over a ten-hour period. Table VIII contains the statistical summary data for both lithium stearate and Ancor GS-6000 trials. The 6s/x, or percent variation, represents the total amount of variation as a percentage of the mean value of that property.

A graph of the O.A.L. production data appears in Figure 20. Clearly, the Ancor 410L with lithium stearate exhibits larger swings in compact O.A.L. than the material with Ancor GS-6000. The percent variation was reduced by 48% when using the Ancor GS-6000, This reduced the need for the operator to make press adjustments during production.

Figure 21 shows the variation in the mass of the compacts during the production comparison. A 29% improvement was experienced when compacting parts with the Ancor GS-6000.

The premix of 410L with Ancor GS-6000 was able to overcome production difficulties associated with part variability and Low green strength, and reduced the green scrap for this application. Non-standard testing of radial crush strength by the parts fabricator indicated that radial crush strength increased by more than 100% using Ancor GS-6000.

CONCLUSIONS

1. Green density and green strength are proportional to the compaction temperature for the range of 75°F to 215°F. Green strength for materials that contain Ancor GS-6000 can reach 7000 psi (48 MPa) at 180°F (82°C), when used with 410L stainless steel.
2. Green expansion is inversely proportional to the compaction temperature. A decrease of 0.1% is achieved by increasing the compaction temperature from 75°F to 215°F.
3. Mixtures of Ancor GS-6000 with lithium stearate (lithium stearate) indicated compromises in green and sintered properties can be achieved, relative to straight lithium stearate or Ancor GS-6000 additions, while realizing the benefits of high green strength. These enables P/M parts fabricators to minimize tooling modifications when switching to the high green strength material.
4. The particle size of the Ancor GS-6000 has a significant effect on sintered properties. Smaller particle size can improve sintered transverse rupture strength and apparent hardness.
5. Production quality problems encountered when using Ancor 410L with lithium stearate were overcome with Ancor GS-6000. The increased green strength eliminated green part failure, while reducing part-to-part variability.

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REFERENCES

1. Cimino, T.M., Luk, S.H., "Machinability Evaluation of Selected High Green Strength P/M Materials", *Advances in Powder Metallurgy & Particulate Materials - 1995*, Part 8, pp 129 -148, Metal Powder Industries Federation, Princeton, NJ.
2. Narasimhan, K.S., "Recent Advances in Ferrous Powder Metallurgy", *Advanced Performance Materials*, V.3, No. 1, Jan 1996, pp 7 -27.
3. Luk, S.H., Davala, A.B., Kopech, H.M., "Enhanced Green Strength Material System for Ferrous and Stainless P/M Processing", *Advances in Powder Metallurgy & Particulate Materials - 1996*, V. 5, Part 17, pp 127 - 142.
4. Klar, E., Shafer, W.M., "On Green Strength and Compressibility in Metal Powder Compaction," *Modern Developments in Powder Metallurgy, 1977*, V. 9, pp 91 -113.
5. Aesoph, M.D., Gurson, A.L., "Relationship between Lubricant Content, Compaction Pressure, Metal Volume Fraction, Bend Strength, and Elastic Modulus in Green Compacts", *Advances in Powder Metallurgy & Particulate Materials - 1994*, V. 3, pp 57 - 69.
6. Armstrong, S., Aesoph, M.D., Gurson, A.L., "The Effects of Lubricant Content and Relative Powder Density on the Elastic, Yield and Failure Behavior of a Compacted Metal Powder", *Advances in Powder Metallurgy & Particulate Materials - 1995*, V. 3, pp 31 - 44.

7. Ball, W.G., Hinger, F.W., McKotch, R.A., "Replacing Internal with External Lubricants", *Advances in Powder Metallurgy & Particulate Materials* - 1994, V. 3, pp 71 - 82.
8. Luk, S.H., Rutz, H.G., Lutz, M.A., "Properties of High Density Ferrous P/M Materials - A Study of Various Processes", *Advances in Powder Metallurgy & Particulate Materials* -1994, V. 5, pp 135 - 154.
9. Bocchini, G.F., Cricri, G., Esposito, R., "Influence of Operating Temperature on the Shrink-Fitting Pressure of P/M Dies", *Advances in Powder Metallurgy & Particulate Materials* -1996, V. 2, Part 7, pp 159 - 178.
10. Metz, P.A., Wolfe, J., German, R., Griffo, A., Steranko, T., "Improved Control of Lubricant Particle Size Distribution and The Effects of Particle Size On P/M Processing", *Advances in Powder Metallurgy & Particulate Materials* - 1996, V. 2, part 6, pp 59 - 71.
11. Zenger, D.C., Cai, H., "Common Cracks in Green P/M Compacts", *Advances in Powder Metallurgy & Particulate Materials* - 1996, V. 7, pp 273 - 280.
12. Reid, J.D., "Avoiding Cracks in the Production of P/M Components", *Metal Powder Report*, Nov., 1986, pp 845 - 849.

Table VI: Effect of Ancor GS-6000 Particle Size on FN-0205 Green Properties

| Material | Pressure (tsi) | Green Density (g/cm ³) | Green Expansion (%) | Green Strength (psi) | Strip Pressure (psi) | Slide Pressure (psi) | Apparent Density (g/cm ³) | Hall Flow (s/50g) |
|----------|----------------|------------------------------------|---------------------|----------------------|----------------------|----------------------|---------------------------------------|-------------------|
| IIIA | 30 | 6.91 | 0.10 | 5500 | 2400 | 1000 | 2.99 | 28 |
| IIIB | 30 | 6.86 | 0.11 | 5400 | 2200 | 900 | 2.94 | 28 |
| IIIC | 30 | 6.86 | 0.10 | 1900 | 2500 | 1200 | 3.09 | 37 |
| IIIA | 40 | 7.17 | 0.12 | 6800 | 2900 | 1100 | 2.99 | 28 |
| IIIB | 40 | 7.09 | 0.14 | 5600 | 3000 | 1200 | 2.94 | 28 |
| IIIC | 40 | 7.05 | 0.14 | 2700 | 2900 | 1100 | 3.09 | 37 |
| IIIA | 50 | 7.24 | 0.16 | 7100 | 3100 | 1100 | 2.99 | 28 |
| IIIB | 50 | 7.18 | 0.17 | 6800 | 2900 | 1100 | 2.94 | 28 |
| IIIC | 50 | 7.13 | 0.17 | 2100 | 3000 | 1600 | 3.09 | 37 |

Table VII: Effect of Ancor GS-6000 Particle Size on FN-0205 Sintered Properties

| Material | Pressure (tsi) | Sintered Density (g/cm ³) | Dimensional Change (%) | Sintered Strength (psi) | Apparent Hardness (HRB) |
|----------|----------------|---------------------------------------|------------------------|-------------------------|-------------------------|
| IIIA | 30 | 6.88 | +0.01 | 110000 | 60 |
| IIIB | 30 | 6.82 | +0.01 | 98000 | 58 |
| IIIC | 30 | 6.86 | -0.06 | 107000 | 60 |
| IIIA | 40 | 7.10 | +0.05 | 121000 | 68 |
| IIIB | 40 | 7.02 | +0.09 | 106000 | 65 |
| IIIC | 40 | 7.02 | +0.03 | 116000 | 63 |
| IIIA | 50 | 7.17 | +0.1 | 114000 | 72 |
| IIIB | 50 | 7.12 | +0.08 | 112000 | 69 |
| IIIC | 50 | 7.13 | -0.03 | 140000 | 70 |

Table I: Premix Compositions

| Material Designation | Material Composition |
|-----------------------------|---|
| IA | 303L+2.25 w/o Cu+1.0 w/o Ancor GS-6000 |
| IB | 303L+2.25 w/o Cu+0.50 w/o lithium stearate+0.50 w/o Ancor GS-6000 |
| IC | 303L+2.25 w/o Cu+1.0 w/o lithium stearate |
| | |
| IIA | 410L+1.0 w/o Ancor GS-6000 |
| IIB | 410L+0.75 w/o Ancor GS-6000 +0.25 w/o lithium stearate |
| IIC | 410L+0.5 w/o Ancor GS-6000 +0.50 w/o lithium stearate |
| IID | 410L+0.25 w/o Ancor GS-6000 +0.75 w/o lithium stearate |
| IIE | 410L+1.0 w/o lithium stearate |
| IIF | 410L+1.0 w/o Ancor GS-6000 (finer than 20 μ m) |
| | |
| IIIA | FN-0205 + 1.0 w/o Ancor GS-6000 (finer than 20 μ m) |
| IIIB | FN-0205 + 1.0 w/o Ancor GS-6000 (regular) |
| IIIC | FN-0205 + 1.0 w/o Acrawax-C |

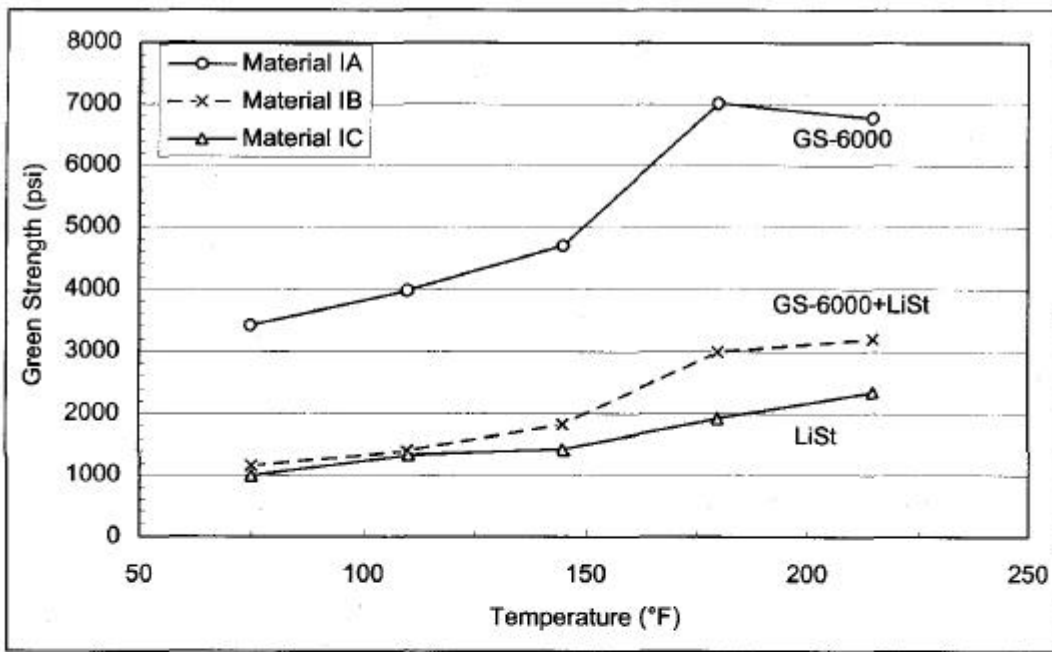


Figure 1: Effect of Temperature on Green Strength at 50 tsi Compaction Pressure

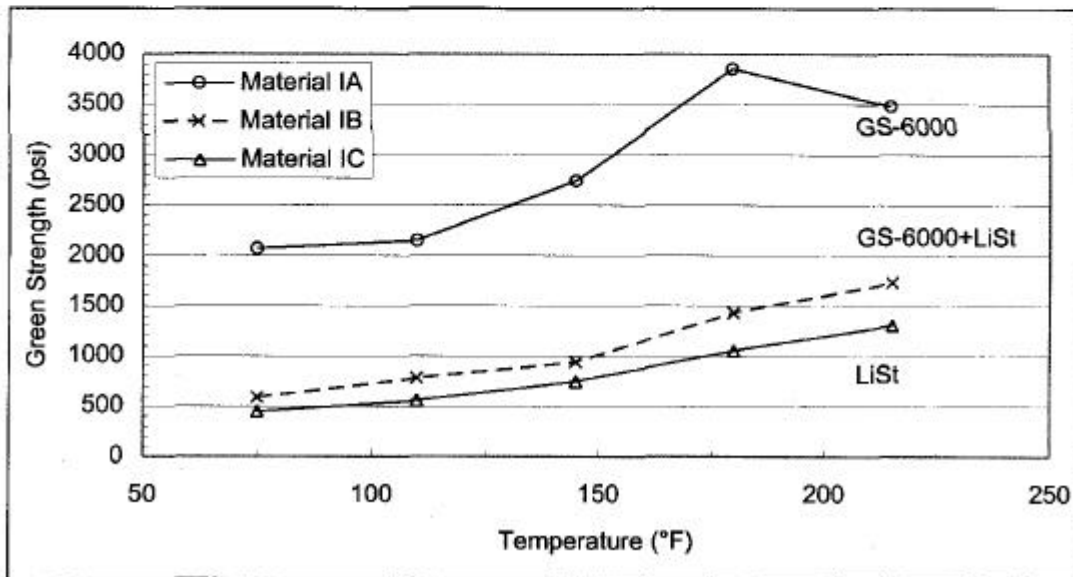


Figure 2: Effect of Temperature on Green Strength at 30 tsi Compaction Pressure

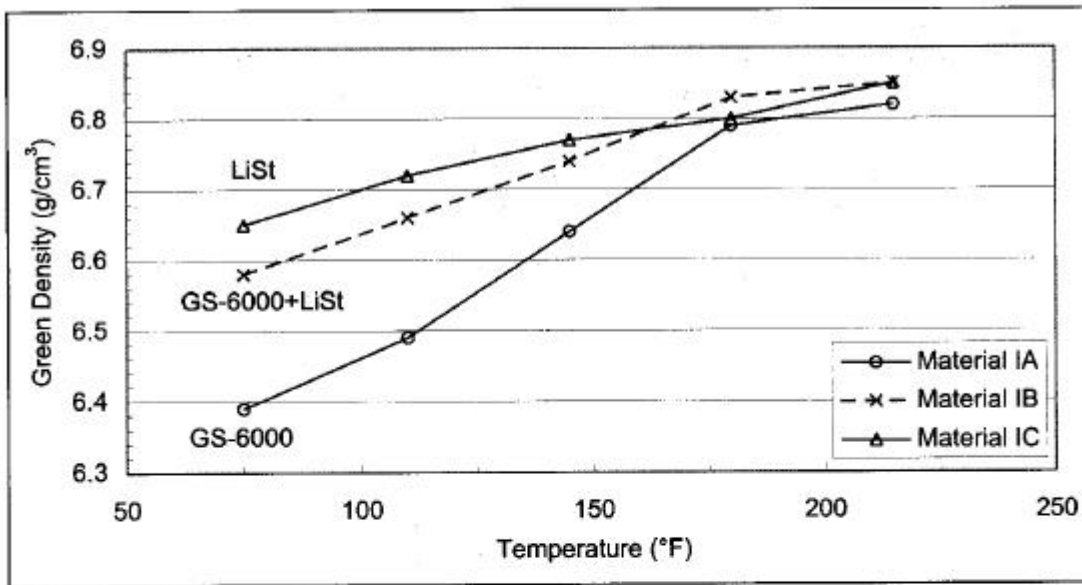


Figure 3: Effect of Temperature on Green Density at 50 tsi Compaction Pressure

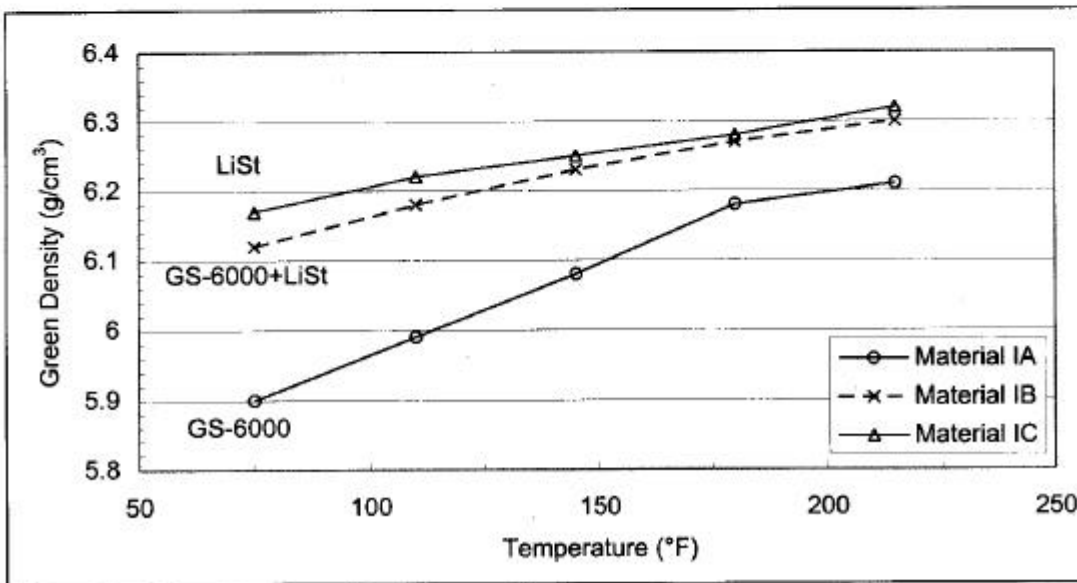


Figure 4: Effect of Temperature on Green Density at 30 tsi Compaction Pressure

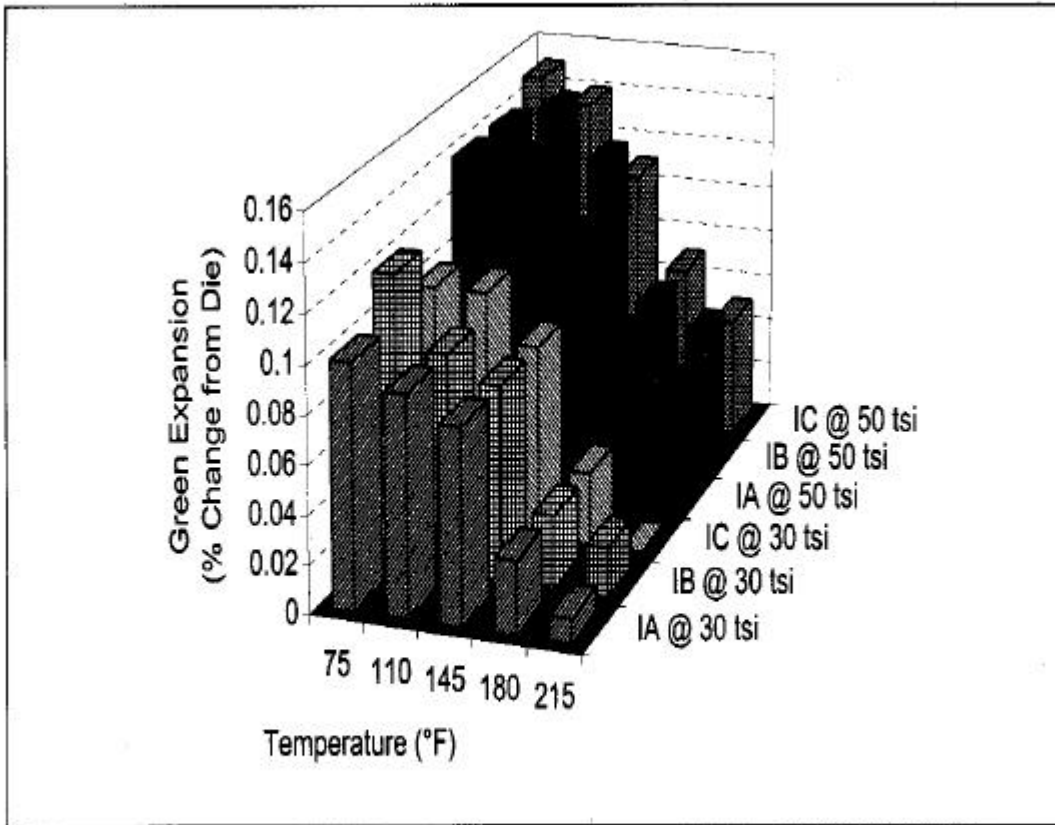


Figure 5: Effect of Temperature on Green Expansion in Direction Normal to the Die Wall at Compaction Pressures Indicated

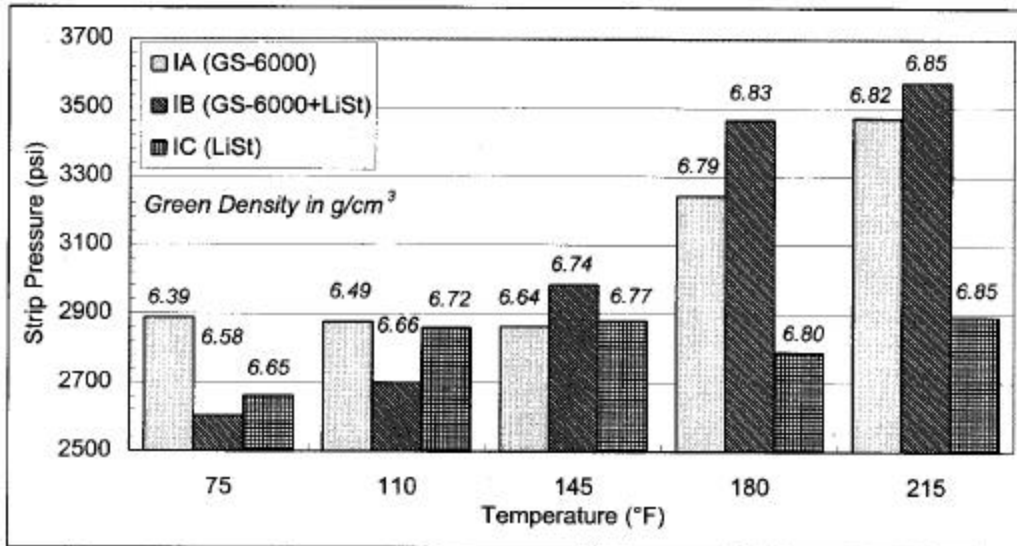


Figure 6: Effect of Temperature on Stripping Pressure at 50 tsi Compaction Pressure

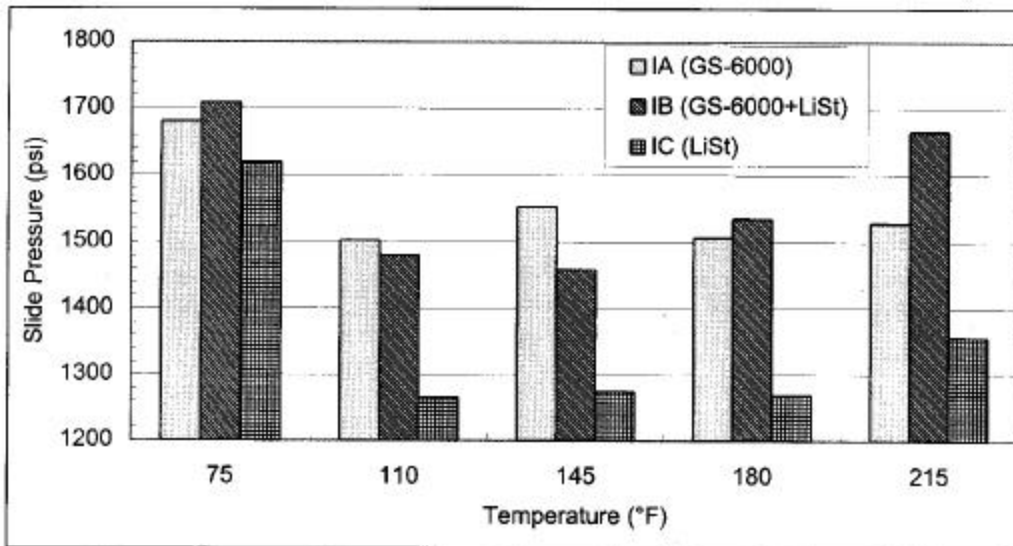


Figure 7: Effect of Temperature on Sliding Pressure at 50 tsi Compaction Pressure

Table II: Effects of Pressing Temperature on Green Properties

| Material | Compaction Pressure (tsi) | Temp (°F) | Green Density (g/cm ³) | Green Strength (psi) | Green Expansion (%) | Strip Pressure (psi) | Slide Pressure (psi) |
|----------|---------------------------|-----------|------------------------------------|----------------------|---------------------|----------------------|----------------------|
| IA | 30 | 75 | 5.90 | 2100 | 0.10 | 1600 | 1000 |
| | | 110 | 5.99 | 2100 | 0.09 | 1600 | 1000 |
| | | 145 | 6.08 | 2700 | 0.08 | 1900 | 900 |
| | | 180 | 6.18 | 3900 | 0.03 | 1900 | 1000 |
| | | 215 | 6.21 | 3500 | 0.01 | 1900 | 1000 |
| IA | 50 | 75 | 6.39 | 3400 | 0.14 | 2900 | 1700 |
| | | 110 | 6.49 | 4000 | 0.14 | 2900 | 1500 |
| | | 145 | 6.64 | 4700 | 0.11 | 2900 | 1600 |
| | | 180 | 6.79 | 7000 | 0.07 | 3200 | 1500 |
| | | 215 | 6.82 | 6800 | 0.05 | 3500 | 1500 |
| IB | 30 | 75 | 6.12 | 600 | 0.12 | 1300 | 900 |
| | | 110 | 6.18 | 800 | 0.09 | 1600 | 800 |
| | | 145 | 6.23 | 900 | 0.08 | 1800 | 900 |
| | | 180 | 6.27 | 1400 | 0.03 | 2000 | 900 |
| | | 215 | 6.30 | 1700 | 0.02 | 1900 | 800 |
| IB | 50 | 75 | 6.58 | 1100 | 0.14 | 2600 | 1700 |
| | | 110 | 6.66 | 1400 | 0.15 | 2700 | 1500 |
| | | 145 | 6.74 | 1800 | 0.13 | 3000 | 1500 |
| | | 180 | 6.83 | 3000 | 0.07 | 3500 | 1500 |
| | | 215 | 6.85 | 3200 | 0.06 | 3600 | 1700 |
| IC | 30 | 75 | 6.17 | 400 | 0.10 | 1500 | 900 |
| | | 110 | 6.22 | 600 | 0.10 | 1600 | 800 |
| | | 145 | 6.25 | 700 | 0.08 | 1800 | 800 |
| | | 180 | 6.28 | 1100 | 0.03 | 1800 | 800 |
| | | 215 | 6.32 | 1300 | 0.00 | 1700 | 700 |
| IC | 50 | 75 | 6.65 | 1000 | 0.15 | 2700 | 1600 |
| | | 110 | 6.72 | 1300 | 0.14 | 2900 | 1300 |
| | | 145 | 6.77 | 1400 | 0.11 | 2900 | 1300 |
| | | 180 | 6.80 | 1900 | 0.07 | 2800 | 1300 |
| | | 215 | 6.85 | 2300 | 0.05 | 2900 | 1400 |

Properties Achieved with a Mixture of Ancor GS-6000 and Lithium Stearate for Ancor 410L Stainless Steel Materials

Table III: Effects of Ancor GS-6000 and Lithium Stearate Mixtures on Green Properties for Ancor 410L Materials

| Material | Pressure (tsi) | Green Density (g/cm ³) | Green Strength (psi) | Green Expansion (%) | Strip Pressure (psi) | Slide Pressure (psi) | Apparent Density (g/cm ³) | Hall-Flow (s/50g) |
|----------|----------------|------------------------------------|----------------------|---------------------|----------------------|----------------------|---------------------------------------|-------------------|
| IIA | 30 | 5.83 | 2200 | 0.15 | 1700 | 900 | 2.71 | 26 |
| IIB | 30 | 5.94 | 1300 | 0.17 | 1800 | 900 | 3.03 | 32 |
| IIC | 30 | 5.97 | 1000 | 0.18 | 1800 | 800 | 3.08 | 38 |
| IID | 30 | 5.97 | 800 | 0.20 | 1600 | 800 | 3.09 | 40 |
| IIE | 30 | 5.98 | 600 | 0.19 | 1700 | 900 | 3.10 | 42 |
| IIA | 50 | 6.47 | 5700 | 0.18 | 3200 | 1500 | 2.71 | 26 |
| IIB | 50 | 6.53 | 3100 | 0.19 | 3400 | 1400 | 3.03 | 32 |
| IIC | 50 | 6.55 | 2100 | 0.19 | 3500 | 1500 | 3.08 | 38 |
| IID | 50 | 6.55 | 1700 | 0.23 | 3200 | 1600 | 3.09 | 40 |
| IIE | 50 | 6.54 | 1500 | 0.22 | 3300 | 2000 | 3.10 | 42 |

Table IV: Effects of Ancor GS-6000 and Lithium Stearate Mixtures on Sintered Properties for Ancor 410 Materials

| Material | Pressure (tsi) | Sintered Density (g/cm ³) | Dimensional Change (%) | Transverse Rupture Strength (psi) | Apparent Hardness (HRB) |
|----------|----------------|---------------------------------------|------------------------|-----------------------------------|-------------------------|
| IIA | 30 | 6.60 | -4.12 | 100000 | 33 |
| IIB | 30 | 6.75 | -4.13 | 110000 | 42 |
| IIC | 30 | 6.76 | -3.99 | 115000 | 43 |
| IID | 30 | 6.82 | -4.20 | 118000 | 43 |
| IIE | 30 | 6.88 | -4.39 | 122000 | 47 |
| IIA | 50 | 7.06 | -3.21 | 126000 | 56 |
| IIB | 50 | 7.18 | -3.21 | 136000 | 62 |
| IIC | 50 | 7.19 | -3.11 | 138000 | 59 |
| IID | 50 | 7.20 | -2.94 | 138000 | 63 |
| IIE | 50 | 7.21 | -3.02 | 154000 | 63 |

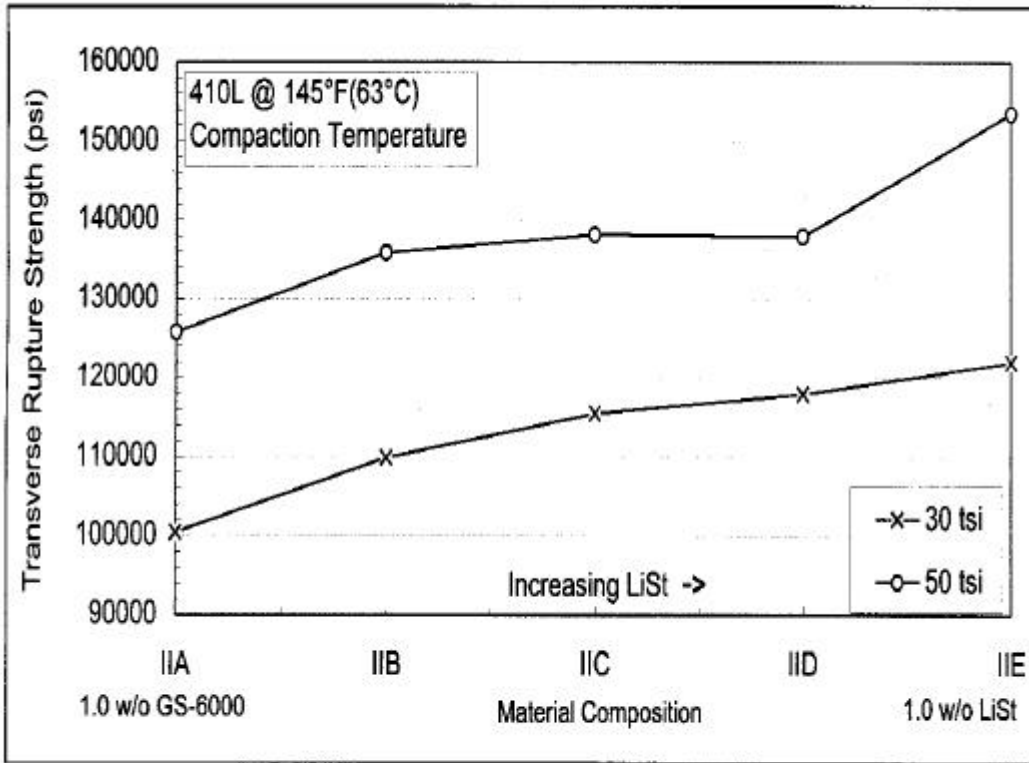


Figure 8: Effect of Lubricant Composition on Sintered TRS of Ancor 410L Materials

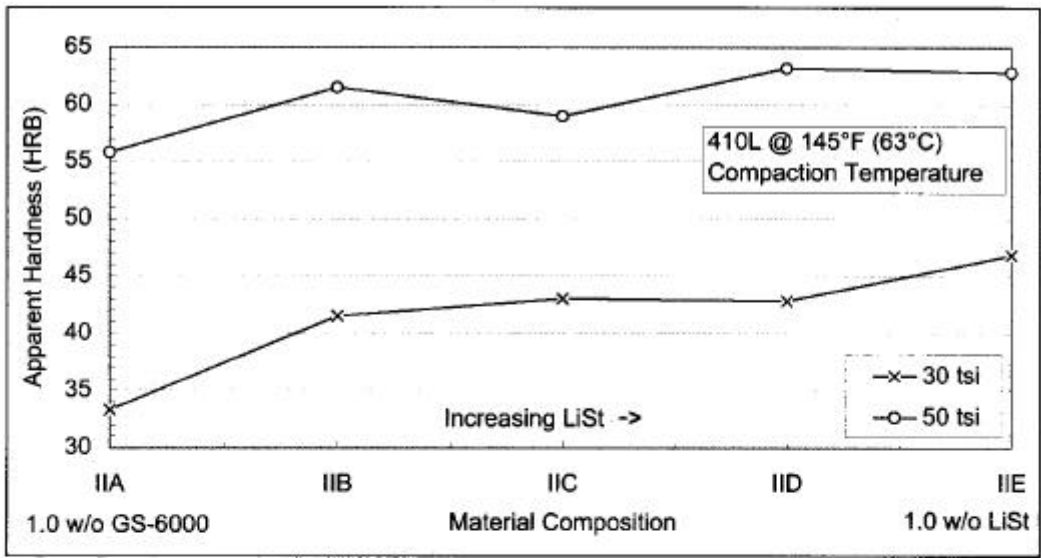


Figure 9: Effect of Lubricant Composition on Apparent Hardness (HRB) for Ancor 410L Materials

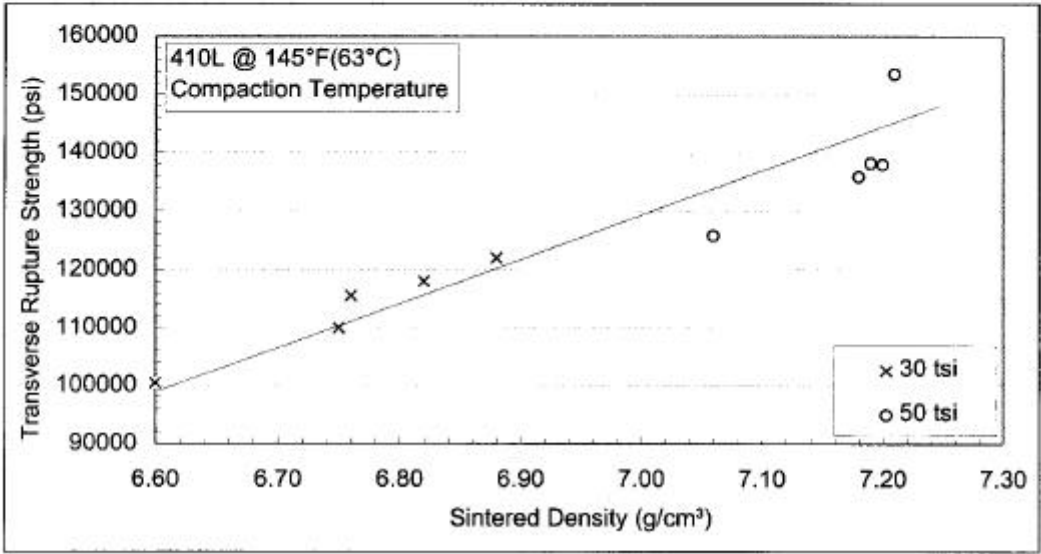


Figure 10: Transverse Rupture Strength as a Function of Sintered Density for Ancor 410L Materials

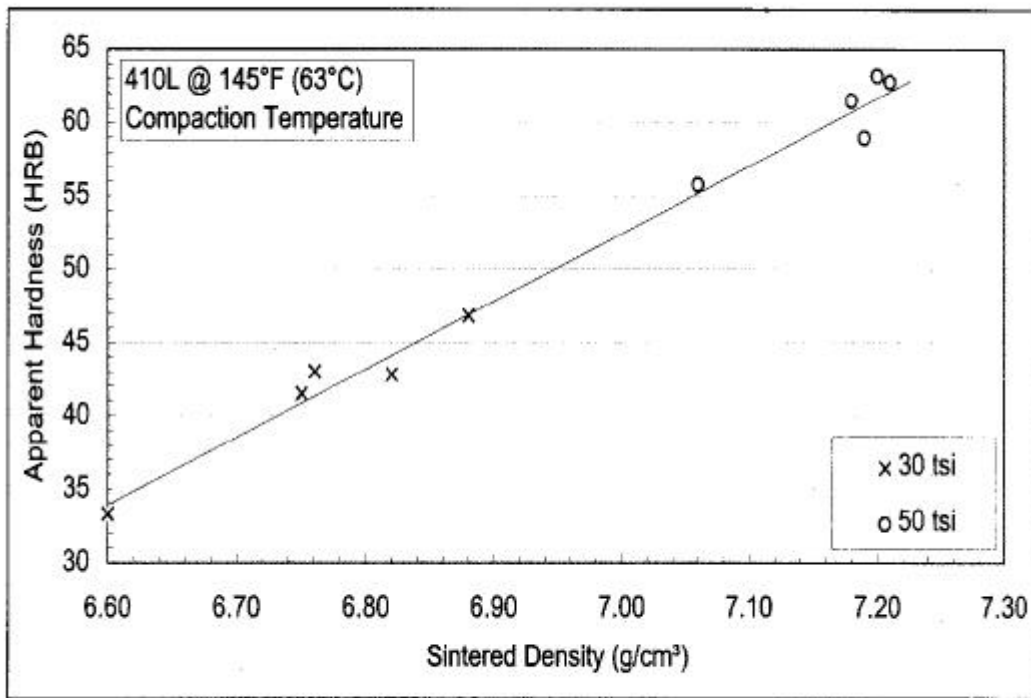


Figure 11: Apparent Hardness as a Function of Sintered Density for Ancor 410L Materials

Table V - Effect of Ancor GS-6000 Particle Size on Ancor 410L Sintered Properties

| Material | Pressure (tsi) | Sintered Density (g/cm ³) | Dimensional Change (%) | Sintered Strength (psi) | Apparent Hardness (HRB) |
|----------|----------------|---------------------------------------|------------------------|-------------------------|-------------------------|
| IIA | 30 | 6.60 | -4.12 | 100000 | 33 |
| IIC | 30 | 6.76 | -3.99 | 115000 | 43 |
| IIE | 30 | 6.88 | -4.39 | 122000 | 47 |
| IIF | 30 | 6.64 | -4.3 | 94000 | 32 |
| IIA | 50 | 7.06 | -3.21 | 126000 | 56 |
| IIC | 50 | 7.19 | -3.11 | 138000 | 59 |
| IIE | 50 | 7.21 | -3.02 | 154000 | 63 |
| IIF | 50 | 7.07 | -3.05 | 138000 | 54 |



Figure 12: Microstructure of Sintered TRS bar (Material IIA)



Figure 13: Microstructure of Sintered TRS bar (Material IIE)



Figure 14: Microstructure of Sintered TRS Bar (Material IIC)



Figure 15: Microstructure of Sintered TRS Bar (Material IIF)



Figure 16: Microstructure of Sintered TRS Bar (composed of 410L with 1 w/o coarse Ancor GS-6000)

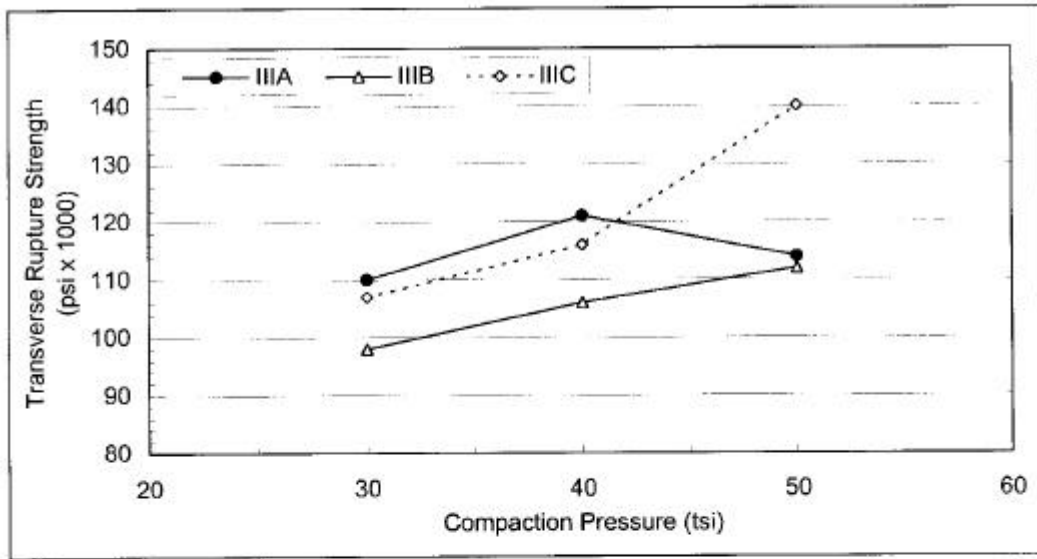


Figure 17: Effect of Ancor GS-6000 Particle Size on TRS of FN-0205

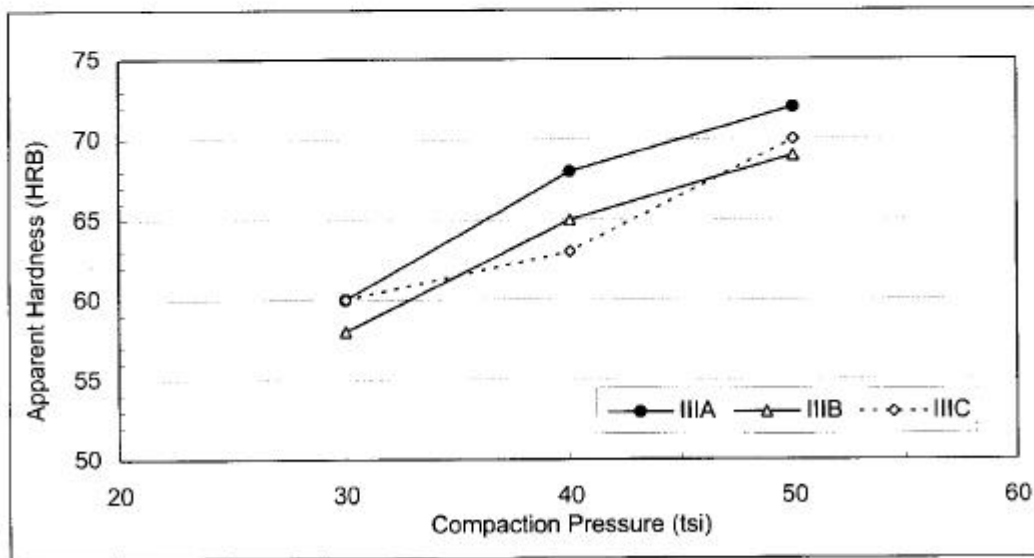


Figure 18: Effect of Ancor GS-6000 Particle Size on Apparent Hardness of FN-0205

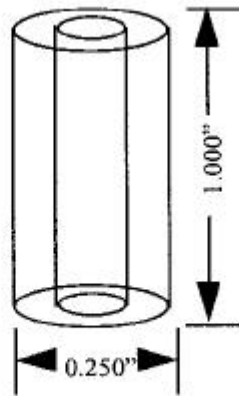


Figure 19: Simplified Illustration of Production Part

Table VIII: Statistical Summary of Production Data

| Composition | Sample Size (n) | Over All Length (O.A.L.) | | | Compact Mass | | |
|-----------------------------|-----------------|--------------------------|---------------|----------|----------------------|---------------|----------|
| | | mean \bar{x} in (mm) | Std. Dev. (s) | 6s/x (%) | mean \bar{x} grams | Std. Dev. (s) | 6s/x (%) |
| + 0.75 w/o Ancor 410L | 220 | 0.9975 (25.31) | 0.0009 | 0.53 | 3.555 | 0.012 | 2.00 |
| + 0.75 w/o lithium stearate | 220 | 1.0003 (25.38) | 0.0017 | 1.02 | 3.551 | 0.017 | 2.82 |