# PM aims for direct competition with 'old-tech' industry

News of the activated nanotech process and the real possibility of achieving full density products caused a stir at PM²TEC2004 in Chicago. So too did the inventors' explicit call for the whole industry to back a push to turn a promising start into a truly disruptive technology that could change the face of metal manufacturing...

While powder metallurgy can produce near net shape components, they usually lack the mechanical properties of wrought products. The industry has attempted to achieve these properties through the use of newly available powders and lubricants, improved pressing techniques and high temperature sintering.

A new process - the activated nanotech process (ANP)* - can dramatically improve the properties and economics of PM and allow powder metallurgy to compete more effectively with machined, wrought and precision-cast components. A patent is pending.

And although further work on fatigue, endurance and thermal treatment remains to be done, it is already clear that the selection of specific lubricants to be used with activated nanotech powders has important effects on pressing characteristics. Among these are maintenance of dimensional control during component production and elimination of "hour glassing".

The secondary operations currently in use do offer improved properties but still have limitations, along with increased costs. As a result, the ferrous PM parts industry is a $2 billion business, a small fraction of the value of the machined wrought material and precision casting component businesses.

Properties offered at the moment by the press and sinter PM industry exhibit densities, tensile strengths, transverse rupture strengths, impact resistance and fatigue properties among others which are, at best, considerably less than their wrought and cast counterparts. Ferrous powder metal parts are, therefore, generally sold as cost-saving components, because they are "good enough" for their defined and limited market.

Ferrous base parts produced with a single press and sinter process can reach a density of 7.1 g/cm³. Secondary operations contribute to higher densities and properties, but they also increase piece part cost. Table 1 identifies the secondary or supplementary operations which may be added and their subsequent density limits.

Powder forging requires very significant capital investment and is limited to the components for which the tooling is designed such as automotive connecting rods. Such investment in tooling and ancillary equipment makes powder forging suitable for production of dedicated components in the many millions, but lacks the flexibility for shorter runs. Process costs in reaching the above densities are shown in Figure 1. The invention of these powders permits the use of the most versatile and least costly method of producing PM parts

* ACTIVATED, NANOTECH and ANP are trademarks of Material Technologies Inc. Patents are pending.

**Table 1**

| Secondary/Supplementary Operation | Max. Developed Density |
|---|---|
| Warm Compaction | 7.3 g/cm |
| Double Press/Double Sinter | 7.4 g/cm |
| High Velocity Compaction | 7.5 g/cm |
| High Velocity Compaction with Double Press/Double Sinter | 7.7 g/cm |
| Hot Forging of PM Preforms | 7.84 g/cm |


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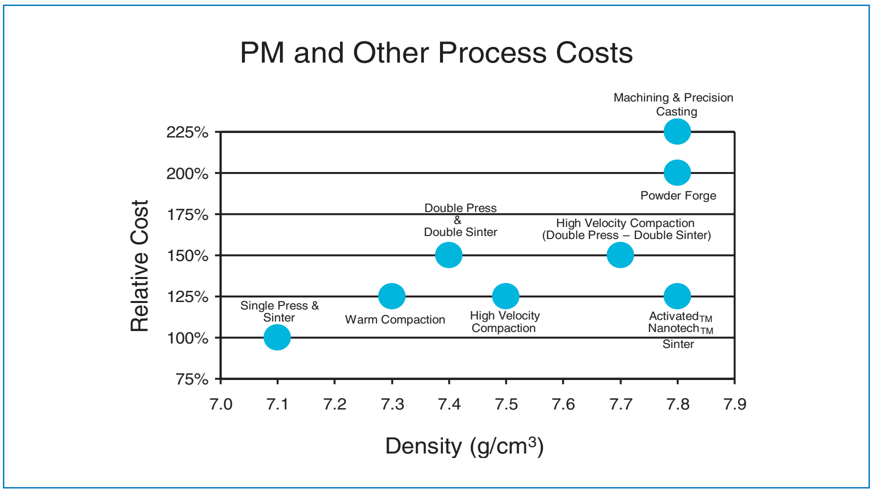



*Figure 1. With cost performance of this order, ANP's future could be assured.*

to make components with equivalent or superior properties to that of wrought steel. While PM has historically been considered a cost-saving production process, there are examples of PM products, which, because of their homogeneity, provide premium products with performance superior to wrought product.

Examples of this are rotating superalloy discs in high-performance jet engines and PM high-speed steels. In this case, PM commands premium prices because of superior, more predictable performance. These powders provide parts manufacturers with the wherewithal to compete in the wrought marketplace, thus offering enormous growth opportunities for the industry.

Certain compositions combined with iron and graphite undergo a complex transient liquid phase at temperatures around 2500oF. These temperatures can be easily obtained with pusher, walking beam or vacuum furnaces that are commonly used in the industry. This metallurgical condition has been reached repeatedly and predictably using Hoeganaes' powder grades 150 HP and 85 HP with nickel contents varying from 2 per cent to 7 per cent. The reaction is enhanced through the use of very fine constituents. Other powder manufacturers' grades have shown similar reactions, but have not been fully tested. Tensile properties of as sintered and heat treated 4 per cent nickel steel can easily reach 1414 MPa as compared to tensile properties of 1030 MPa for a similar conventionally pressed, sintered and heat treated composition.

The full density achieved by as-sintered parts produced using this technology, allows the elimination of secondary steps such as impregnation, which is a considerable manufacturing cost. Furthermore, such dense parts can be heat treated by methods that are suitable for wrought materials thus further enhancing the properties of such dense PM parts.

Dimensional control is extremely precise and surface condition is excellent.

## The role of lubricants

The use of specific lubricants has assisted in pressing these materials at pressures ranging from 20 tons to 60 tons per square inch (tsi). Such materials compacted under these pressures have reached full densities after sintering.

APEX Advanced Technologies, LLC (APEX) has developed an advanced lubricant which has numerous benefits that facilitate achieving uniform near full density from water atomised metal in a press and sinter process.

Higher green density is the first step toward near full density parts. To achieve greater green densities with conventional single press and sinter processes, the following critical criteria must be met:

- The level of lubricant used must be reduced while not negatively impacting the process or part;
- The lubricant must be more effective;
- The lubricant must be mobile so it migrates during pressing;
- The lubricant must be effectively removed during the sintering process; and
- In addition, the lubricant must not introduce other negative characteristics such as environmental issues, increased processing time or reduced yields.

Increased effectiveness and a reduced level of lubricant are both directly a result of mobility. Mobility allows the lubricant to migrate to the die wall, which produces the effectiveness and allows for a reduced use level. Reduced use level provides a lower volume of organic material, and therefore higher densities.

Such lubricants transform from solid to viscous liquid with pressure and shear at room temperature. This is a very abnormal phenomenon and is unique to certain

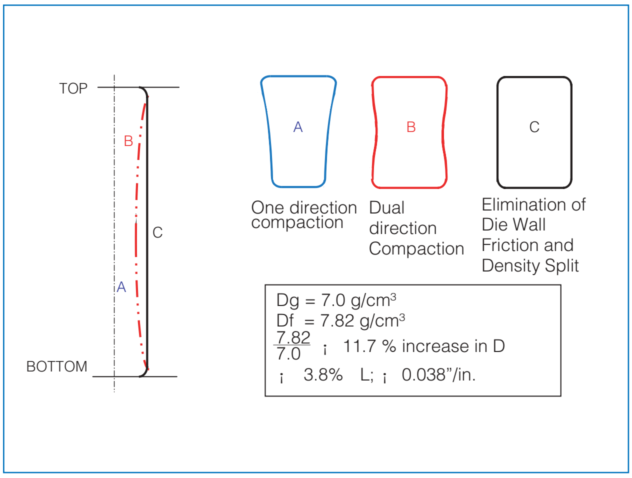



*Figure 2. The lubricant used helps minimise press distortion.*

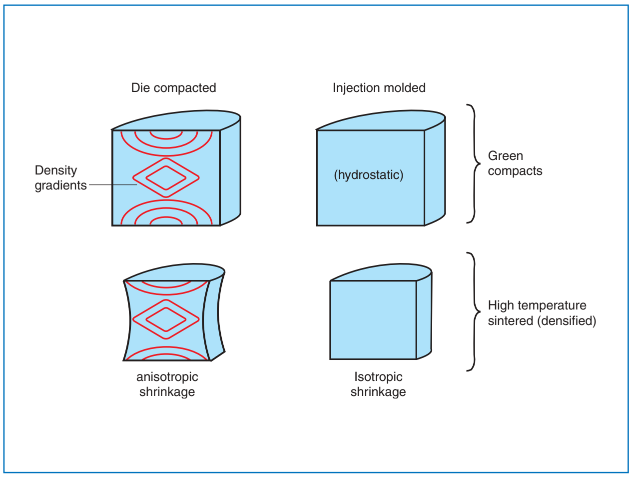
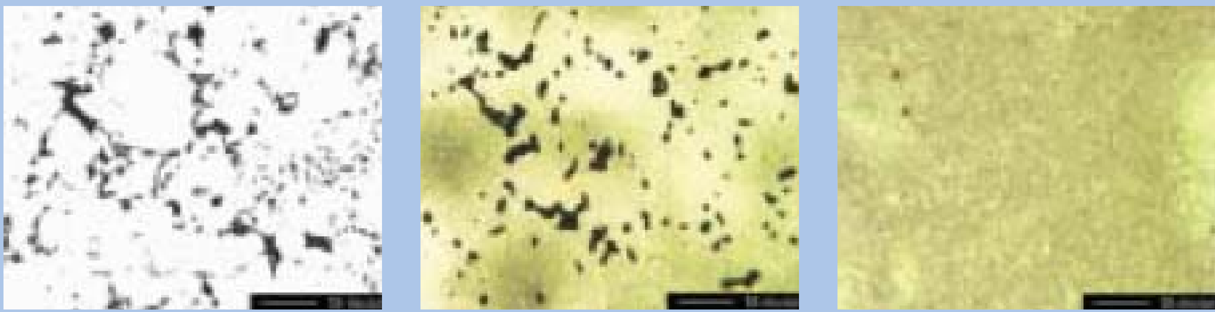
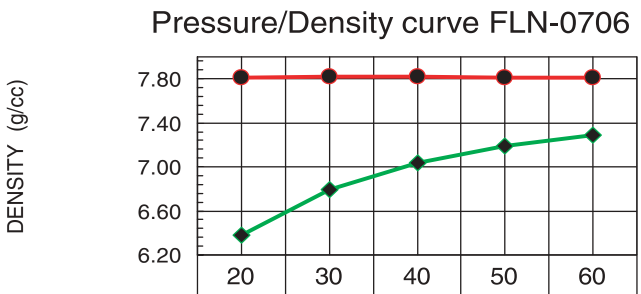



*Figure 3. Greater green density increases precision in sintering.*



| Standard PM | High Density PM | ANP™ |
|---|---|---|
| 6.70 g/cc | 7.41 g/cc | 7.84 g/cc |
| (85% Theoretical Density) | (95% Theoretical Density) | (→99.5% Theoretical Density) |

*Figure 4. Comparison of Microstructures.*




| Mpa | 276 | 414 | 552 | 690 | 828 |
|---|---|---|---|---|---|
| Green | 6.38 | 6.79 | 7.04 | 7.19 | 7.29 |
| Sintered | 7.81 | 7.82 | 7.82 | 7.81 | 7.81 |

*Figure 5. Pressed and Sintered Pressure/Density curves*

## A task for the industry

ANP materials produce part properties comparable and potentially superior to wrought products due to reduced impurity levels, say its inventors Richard Phillips and Ira Friedman.

This technology is disruptive to other manufacturing technologies such as precision casting and the machining of bar stock, but it will be most disruptive in a shorter amount of time if the entire PM industry makes a concerted effort to commercialise such products. Obvious beneficiaries are the powder producers, tool makers, press manufacturers, furnace manufacturers, parts makers, heat treat shops, machine shops and other participants across the entire industry.

This is an activity which can and should be promoted by the entire industry and the MPIF since it involves education of the ultimate consumers who need to understand that this is a high-performance product which should be sold based upon its superior properties as well as dramatic cost savings.

Development efforts are underway to achieve relatively high densities at lower sintering temperatures, but the industry innovators will need to adapt high temperature sintering which is available on a toll basis until parts producers achieve the critical mass to warrant investment in such furnaces.

The vast machined and cast part markets are there for PM penetration, but development work such as property quantification and investment in high-temperature sintering capacity for this technology is needed for such disruption to take place on a very large scale.

lubricants. When the lubricant is pressed, it is transformed to a liquid at about 4tsi at room temperature. This allows for rearrangement of the metal particles to obtain a better fit before they begin to interlock.

This property allows for an immediate improvement in density. The ability of this to occur at room temperature produces a good fit with press operations. As the lubricant transforms from a solid to a liquid it seeks open porosity. As porosity is closed by the press operation the liquid moves to the die wall. The lack of mobility with conventional lubricants often causes site defects such as delaminating and cracking.

Upon liquefaction the lubricant moves to the die wall, reducing frictional resistance and eliminating density split. This liquefaction causes a hydrostatic environment within the compact and reduces green density variation, (Figures 2 & 3). The resultant shrinkage is isotropic, minimising distortion and increasing dimensional precision or a lower coefficient of dimensional variance.

All lubricants must be removed from the part during the sintering operation with minimal negative impact on the part. As porosity is closed, removal of the lubricant becomes more difficult. Ideally, the lubricant will exit the part gradually, so it will not cause problems such as blisters, cracks, blowouts, etc. The use of the appropriate lubricant has the following advantages in the sintering process:

- Since a lower level of lubricant is required, less lubricant will need to exit the part reducing the level of potential problems;

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- The lubricant begins to exit the part at lower temperatures than do the typically utilised grades and exits the part more gradually during the sintering process; and
- The lubricant migrates to the die wall, further reducing the amount of lubricant that needs to be eliminated from the part.

### Excellent dimensional stability

This unique lubricant used for near full-density parts has several desirable attributes.

- Due to the mobility of the lubricant, metal particles fit better making the green part relatively stress free;
- Pores are small and uniform throughout the green part;
- Press tonnage can be increased without micro cracking and delamination;
- Lubricant removal is gradual due to reduced level of lubricant and an extended decomposition range;
- The effectiveness of the lubricant allows for no unplanned density gradients in the green part. Density split is eliminated with the appropriate Apex lubricant;
- Resulting parts are dimensionally stable during sintering and shrink uniformly and predictably; and
- Enhanced sintering efficiency is experienced.

The lubricant used with these powders is a unique organic lubricant that has the following key attributes.

- The lubricant is attracted and attaches to metal surfaces; and
- The lubricant transforms from a solid to a liquid with shear, at lower pressure and temperatures.

As a result;

- Metal particles rearrange at a lower tonnage to obtain better fit;
- The amount of lubricant is reduced;
- Lubricant is mobile within the part eliminating micro cracking and delamination;
- Lubricant migrates to the die wall, allowing uniform porosity and improved green density;
- Rate of gas evolution is decreased due to reduced level of lubricant and more gradual decomposition;
- Press tonnage can be increased without negative impact;
- Dimensional stability is excellent and shrinkage is predictable; and
- Sintering is enhanced

The base materials for this processing were Hoeganaes Ancorsteel 85HP and 150HP formulated to provide a resulting alloy with 0.56 per cent carbon and nickel contents between 2 per cent and 6.6 per cent. The materials were pressed at 20tsi, 30tsi, 40tsi, 50tsi and 60tsi.

These materials were compacted and sintered into transverse rupture specimens (TRS), tensile bars and impact bars to measure density, ultimate tensile strength, yield strength, elongation, and modulus properties. The tensile properties were collected from both compacted bars and machined bars via MPIF [2], and ASTM standards.

Table 2 lists the tests and standard test methods used to collect the material property data.

The green samples were sintered in a pusher furnace with a nitrogen/hydrogen

**Table 2: Test Methods.**

| Standard | MPIF | ASTM | Comments |
|---|---|---|---|
| Density | 42 | B328 | Modified Method |
| Hardness Macro | 43 | E18 | |
| Impact Notched | | E23 | |
| Impact Un-notched | 40 | E23 | |
| Modulus of Elasticity | 10 | E8 | |
| Ultimate Tensile Strength | 10 | E8 | |
| Yield Strength | 10 | E8 | |
| Elongation | 10 | E8 | |
| Microstructure | | E3 | |
| Photomicrographs | | E883 | |

**Table 3: Tensile Data**

| Sample Type | Density, g/cc | UTS, MPa ($10^3$ psi) | 0.2% YS, MPa ($10^3$ psi) | %Elong. | % ROA | Mod of Elas., GPa ($10^6$ psi) | Hardness HRC |
|---|---|---|---|---|---|---|---|
| Pressed | 7.81 | 1,486 (215) | 1.296 (187) | 3.1 | | 188 (27.3) | 43.5 |
| Machined | 7.79 | 1,383 (200) | 1,304 (189) | 2.9 | 9.34 | 182 (26.4) | 45.0 |
| Pressed | 7.59 | 1,446 (209) | 1,205 (175) | 4.0 | | 158 (22.9) | 39.7 |
| Machined | 7.57 | 1,414 (205) | 1,248 (181) | 4.2 | 10.48 | 162 (23.5) | 38.7 |
| Pressed | 7.82 | 1,182 (171) | 955 (138) | 5.2 | | 183 (26.6) | 34.3 |
| Machined | 7.79 | 987 (143) | 939 (136) | 3.5 | 11.51 | 192 (27.8) | 35.4 |
| Pressed | 7.46 | 1,051 (152) | 685 (99) | 4.4 | | 141 (20.4) | 28.0 |
| Machined | 7.57 | 1,314 (190) | 1,211 (176) | 4.9 | 15.92 | 164 (23.8) | 37.5 |
| Pressed | 7.76 | 1,062 (154) | 803 (116) | 8.6 | | 170 (24.6) | 32.0 |
| Machined | 7.79 | 1,036 (150) | 787 (114) | 4.2 | 13.48 | 171 (24.8) | 32.6 |
| Pressed | 7.60 | 1,188 (172) | 690 (100) | 4.0 | | 136 (19.8) | 36.7 |
| Machined | 7.57 | 1,089 (158) | 730 (106) | 7.4 | 15.19 | 135 (19.6) | 32.0 |

**Table 4: Comparison to Wrought Tensile Data 1**

| Grade | ULT MPa ($10^3$ psi) | 0.2% Y.S. MPa ($10^3$ psi) | % Elongation | Hardness, HRC Scale |
|---|---|---|---|---|
| ANP™ FLN-0706 Range of Data | 1,486 (215.4) 1,383 (200.5) | 1,296 (187.9) 1,304 (189.1) | 3.1/2.9 | 43.5/45.0 |
| AISI 4140 | 1,449 (210) | 1,346 (195) | 14 | 45 |
| AISI 4340 | 1,449 (210) | 1,325 (192) | 14 | 45 |
| AISI 5140 | 1,304 (189) | 1,228 (178) | 14 | 40 |
| AISI 4150 | 1,573 (228) | 1,484 (215) | 9 | 47 |
| AISI 5150 | 1,435 (208) | 1,346 (195) | 11 | 45 |
| AISI 6150 | 1,401 (203) | 1,325 (192) | 10 | 46 |

**Table 5: Comparison to Wrought Tensile Data 2**

| Grade | ULT MPa ($10^3$ psi) | 0.2% Y.S. MPa ($10^3$ psi) | % Elongation | Hardness, HRC Scale |
|---|---|---|---|---|
| ANP™ FLN-0706 Range of Data | 1,182 (171.3) 987 (143.1) | 955 (138.4) 939 (136.1) | 5.2/3.5 | 34.3/35.4 |
| AISI 4140 | 1,021 (148) | 917 (133) | 18 | 33 |
| AISI 4340 | 1,049 (152) | 979.8 (142) | 18 | 34 |
| AISI 5140 | 911 (132) | 800 (116) | 20 | 28 |
| AISI 4150 | 1,242 (180) | 1,118 (162) | 12 | 39 |
| AISI 5150 | 980 (142) | 911 (132) | 18 | 31 |
| AISI 6150 | 1,125 (163) | 1,063 (154) | 15 | 36 |

**Table 7: Charpy Impact Comparison**

| Density, g/cc | FLN-0706 (No Notch), ft-lbf | FLN-0706 (Notched), ft-lbf | Wrought (Notched), fl-lbf |
|---|---|---|---|
| 7.8 | 50 | 7 | 12-17 |
| 7.6 | 55 | 6 | |
| 7.8 | 77 | 13 | 36-56 |
| 7.6 | 67 | 11 | |
| 7.8 | 84 | | 77-87 |
| 7.6 | 70 | | |

atmosphere and at a temperature near 2500°F. All samples were subsequently heat treated and tempered similar to wrought steel to produce selective combinations of properties.

The microstructure of the near full density ANP is shown in Figure 4 compared to the lower density standard PM microstructures. The ANP photomicrograph shows a fully hardened martensitic microstructure with some small uniform spherical porosity.

The sintering characteristics of these powders are shown in Figure 5. Both low green density and high green density compacts achieve full density during sintering while maintaining good size control.

The typical dimensional tolerance for standard PM processing is <0.2 per cent compared to ANP findings of 0.17 per cent in the press direction and 0.084 per cent in the perpendicular to the pressed direction. The typical linear size change at 552 MPa (40 tsi) from die size to final product is -3.8 per cent and 2.8 per cent for final sintered densities of 7.82 g/cm³ and 7.60 g/cm³ respectively.

Tensile properties were generated from pressed tensile bars and machined tensile bars. Two density groups and three manufacturing conditions were used in the design of experiments. The tensile data is displayed in Figure 6. These properties were developed utilising the sequence of moulding, sintering, and heat treating method. Figures 7 to 9 are comparisons of ANP tensile properties to low alloy wrought properties developed with the same processing conditions. These materials responded with the similar values for ultimate tensile strength, yield strength, and hardness.

ANP charpy impact bars were measured in the no notch and notched conditions. The resulting data was compared to notched low alloy wrought steel in Figure 10. Typical charpy values for low alloy intermediate PM materials range from 12ft-lbf to 14 ft-lbf in the no notched condition [3]. Such properties are a factor of four to six times higher than intermediate PM materials. The higher no notch values of this material allow the consideration of measuring PM impact properties in the notched condition.

## The authors

THIS article was taken from *Full-density, as-sintered powdered metal (PM) parts produced from water-atomised powder with properties comparable to wrought steel*, a paper presented at PM²TEC2004 in Chicago under the auspices of the Metal Powder Industries Federation. It was prepared by Richard R Phillips of Engineered Pressed Materials and Dennis Hammond of Apex Advanced Technologies who, together with Ira L Friedman of Material Technologies Inc, are collaborators in the development of the ANP process, its associated powders and lubricants.

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## Conclusions

- ANP processing of -100 mesh ferrous powder alloys creates material properties similar to those of wrought product.
- It uses conventional blending and PM moulding capabilities.
- ANP is activated during high-temperature sintering resulting in densification.
- Dimensional control in ANP production is predictable and uniform.
- ANP can use conventional wrought metal processing to meet specific engineering design requirements to enhance optimum product performance.
- ANP parts can be pressed from 276 (20) to 828 (60) MPa (tsi) and still sinter to full density.
- It is expected that further development work will result in even greater performance in ANP materials based upon the potential for further improvement through both material and process modifications. Anomalies and directionality in wrought product which do not exist in fine-grained, homogenous PM parts, should allow activated nanotech powders and parts to provide superior performance to wrought products.

## Acknowledgements

THE authors would like to thank Product Assurance Laboratories of Ridgway, Pennsylvania, for their many hours of lab work and advice in support of this work, as well as Hoeganaes Corporation for providing production lots of powder and pioneering customers who are now developing the significant competitive advantages of this powder. They would also like to thank Material Technologies, Inc for funding this development work and providing manufacturing, intellectual property and marketing support. They would like to thank Al Dornisch, Ron Posteraro, Don Walker, and Crucible Research for their contributions to this programme.

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