LEAN DIFFUSION ALLOYED STEEL FOR HEAT TREATED APPLICATIONS

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ABSTRACT

While recently a large effort has been made to utilize sinter hardening for high performance components, a sizable portion still require the tradition quench and temper heat treatment process. Heat treatable alloys often contain high amounts of copper, molybdenum and nickel to achieve proper hardenability. With recent fluctuations in metal prices, these alloys have come under pressure from competing technologies. A newly developed lean diffusion alloyed material, D.AQ, has been developed to combine excellent properties in the heat treated condition with high compressibility, high workability and improved machinability in the sintered condition while maintaining the trademark stability and robustness of diffusion alloying. This paper will compare the newly developed diffusion alloyed material with other common PM heat treat alloys.

INTRODUCTION

Heat treatment is used to tailor the phase distribution in the microstructure and the distribution of alloying elements after sintering of a powder metal (PM) component.[1] By adjusting alloying additions & cooling rates, a variety of microstructures and performance characteristics can be developed. The heat treatment of PM components can be carried out by a traditional austenitize, quench and temper process or a sinter hardening process.

Heat treatments utilizing the austenitize, quench and temper concept have been in existence for thousands of years. These methods, developed by the wrought and casting industries, can be used on PM components with slight adjustments to account for the inherent porosity of PM. The porosity of a PM component not only reduces the thermal conductivity but also the apparent hardness of the material.[2] The porosity also effects the hardenability. Interconnected porosity will allow the gaseous heat treatment media to penetrate the PM component. This is especially important in carburize, quench and temper operations where the objective is to provide a narrow hardened case on the surface.

The sinter hardening method has become an established manufacturing process to achieve the hardness and strength requirements in the powder metal industry over the last 15 years.[3] Traditionally these alloys contained high amounts of nickel, manganese and molybdenum in order to transform completely to martensite during cooling. With increasing pressure on metal prices and recent advances in convective cooling units, leaner alloys have been developed to allow for a similar metallurgical response to the traditional alloys.[4] While these alloys are leaner compared to the traditional alloys, they still contain a large amount of alloying in order to have a martensitic transformation at typical sinter hardening cooling rates (2 – 3 °C/s).

Due to the differences in cooling rates utilized by conventional heat treat and sinter hardening, conventional heat treatment does not need the alloying levels of sinter hardening alloys. Sinter hardening alloys typically require higher carbon contents to achieve the necessary hardenability. The high carbon content tends to reduce the mechanical properties attainable after hardening.[5] Besides the difference in carbon levels needed, sinter hardening alloys also utilize a copper addition to increase the hardenability.[6] Heat treat alloys, due to the higher cooling rate, are able to avoid these increased alloying levels.

Regardless of the hardening method chosen, dimensional consistency of the as heat treated component is an important characteristic of the material system. If the dimensional consistency is poor, often a machining operation is needed to meet tolerance demands. These machining operations can be difficult and are avoided if possible.[7] Once the production and heat treat processes are stabilized, any inconsistency in dimensions will be related to the alloying method. Admixed material systems are prone to segregation due the constituents' different particle morphology, density, and size distribution. One option to reduce segregation is to add the alloying elements in the melt and atomized. While this eliminates segregation, the result in a less compressible powder due to the alloying in the iron matrix.[8] Another option is to diffusion alloy the elements to the iron particle. This method eliminates segregation while preserving the compressibility of the iron particle. Diffusion alloyed materials are known worldwide for their robustness and dimensional consistency.[9]

A newly developed diffusion alloy, D.AQ, was designed to use the advantages of the diffusion alloy process to produce a leaner heat treat alloy. In this paper, the new alloy is compared against other common heat treat alloys. Static properties and dimensional consistency are compared as sintered and as heat treated.

EXPERIMENTAL PROCEDURE

Material Systems

Several commercially available PM alloys used in the heat treatment of PM components were compared to a newly developed diffusion alloyed base iron, D.AQ. The alloys selected are listed in Table 1.

Table 1. Nominal Compositions of Alloys (wt %)

Base Iron	Mo	Ni	Cu
D.AE	0.50[1]	4.0[1]	1.50[1]
Astaloy 45Mo	0.45[2]	-	-
D.AQ	0.50[1]	0.50[1]	-

1. Diffusion alloyed 2. Pre-alloyed

These base irons were mixed with graphite, lubricant, and / or nickel to create commonly heat treated material system. Natural graphite (SW-1651, Asbury) nickel (Type 123, Inco) and amide wax (Acrawax C, Lonza) were used in the mixes. The compositions of the mixes are shown in Table 2.

Table 2. Nominal Mix Compositions (wt %)

MPIF Designation	Base Iron	Ni	Graphite	Lubricant
N/A	D.AQ	-	0.6	0.75
FD-0405	D.AE	-	0.6	0.75
FLN2-4005	Astaloy 45 Mo	2	0.6	0.75
FL-4005	Astaloy 45 Mo	-	0.6	0.75

Mechanical Property Specimens

From each mix, specimens for tensile strength (MPIF Std. 10), charpy impact energy (MPIF Std. 40), and transverse rupture specimens (TRS) (MPIF Std. 41) were compacted to a green density of 7.00 g/cm^3 using a 60 ton hydraulic compaction press.[10-12] The specimens were sintered in a 6 inch Abbott mesh belt furnace according to the sintering conditions listed in Table 3. Heat treatment was performed according to the parameters listed in Table 4 by Pennsylvania Industrial Heat Treaters (St. Mary's, Pennsylvania). After heat treating, tensile and impact testing was performed according to ASTM E8 and ASTM E23 by Westmoreland Mechanical Testing & Research (Youngstown, Pennsylvania).[13,14] Transverse rupture testing was conducted according to MPIF Standard 41.[12] The specimens were also evaluated for apparent hardness (MPIF Std. 43) and sintered carbon according to ASTM E1019.[15,16]

Table 3. Sintering Conditions

Sintering Temperature	1120 °C (2048 °F)
Time at Temperature	30 minutes
Atmosphere	90 v/o N_2 / 10 v/o H_2
Cooling Rate	0.5 °C/s (1.0 °F/s)

Table 4. Heat Treatment Parameters

Type	Batch
Temperature	921 °C (1690 °F)
Carbon Potential	0.6%
Soak Time	20 minutes
Atmosphere	Endothermic
Quenching	Oil 60 °C (140°F)
Tempering	200 °C (392 °F)
Temper time	1 hour
Temper atmosphere	Air

Hardenability& Mass Effect Specimens

Large rectangular compacts were compacted to a green density of 7.00 g/cm^3 for each mix courtesy of Cavity Masters (Chicago, IL). The compacts were sintered according to the parameters listed in Table 3. After sintering, the compacts were sectioned into blanks which were then machined into the specified dimensions of 100 mm (4 inch) length by 25 mm (1 inch) diameter. Hardenability was evaluated using the Jominy end-quench method according to ASTM Standard A 255 and MPIF Standard 65.[17,18] The samples were austenitized at 926 °C (1700 °F) for 30 minutes in a 90 v/o nitrogen 10 v/o hydrogen atmosphere. The samples were then water end – quenched.

Two different size cylinders were compacted to determine the effect of mass on each material system. 25 mm (1 inch) diameter by 25 mm (1 inch) length cylinders were compacted to a green density of 7.00 g/cm^3 using a Gasbarre (Dubois, PA) 60 ton hydraulic compaction press. 50 mm (2 inch) diameter by 50 mm (2 inch) length cylinder were compacted to a green density of 7.00 g/cm^3 by Cavity Masters. The cylinders were sintered and heat treated according to the parameters in Tables 3 and 4 respectively. After heat treatment, metallographic investigation was performed to characterize the phases present starting at the surface and moving toward the center of the specimens. Microindentation hardness readings were performed according to MPIF Standard 51.[19]

Dimensional Stability Study

A dimensional stability study was conducted on ring specimens with dimensions of 55 mm (2.17 inch) outer diameter by 35 mm (1.38 inch) inner diameter by 10 mm (0.39 inch) height. The ring specimens were compacted from each mix to a green density of 7.00 g/cm^3 on a 100 ton mechanical Gasbarre compaction press. The rings were sintered and heat treated according to the conditions listed in Tables 3 and 4. The outer diameter was measured using a Unite-A-Matic 8600 Gear Inspection machine (Unite-A-Matic, Cincinatti, Ohio). Measurements were taken after sintering and after heat treatment. The out of roundness was calculated according to Equation 1. One measurement was taken in the filling direction, while the other was taken perpendicular to the filling direction. A schematic of the OOR measurement method is presented in Figure 1.

Equation 1. ***Out of Roundness = Absolute Value (D1-D2)***

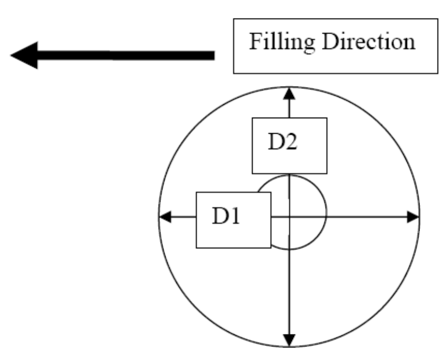

Figure 1. Out of roundness measurement schematic.

Metallography

Metallographic investigations were performed on the as sintered as well as heat treated samples. The polishing procedures and equipment are listed in Table 5 (as sintered) and Table 6 (heat treated). Struers (Copenhagen, Denmark) equipment and consumables were utilized in the preparation of the specimens. The etchants used for each material are listed in Table 7.

Table 5. As Sintered Metallographic Steps / Equipment

Step	Equipment	Wheel / Pad	Media
Cutting	Discotom-5	40A25 Cutoff Wheel	Water Coolant
Mounting	LaboPress-3	N/A	N/A
Grinding	Abraplan	Aluminum Oxide Stone	Water Coolant
Fine Grinding	RotoForce-4 / RotoPol-22	MD Allegro Pad	9 µm Diamond Dosing
Polishing 3 µm	RotoForce-4 / RotoPol-22	MD MOL Pad	3 µm Diamond Dosing
Polishing 1 µm	RotoForce-4 / RotoPol-22	MD NAP Pad	1 µm Diamond Dosing

Table 6. As Heat Treated Metallographic Steps/ Equipment

Step	Equipment	Wheel / Pad	Media
Cutting	Discotom-5	60A25 Cutoff Wheel	Water Coolant
Mounting	LaboPress-3	N/A	N/A
Grinding	Abraplan	Aluminum Oxide Stone	Water Coolant
Fine Grinding	RotoForce-4 / RotoPol-22	MD Allegro Pad	9 µm Diamond Dosing
Polishing 3 µm	RotoForce-4 / RotoPol-22	MD MOL Pad	3 µm Diamond Dosing
Polishing 1 µm	RotoForce-4 / RotoPol-22	MD NAP Pad	1 µm Diamond Dosing

Table 7. Etchant Selection

Material	Etchant
New Alloy	2% Picral
FD-0405	2% Picral
FLN2-4005	1% Nital / Picral
FL-4005	1% Nital / Picral

RESULTS

Mechanical Properties

The results of the tensile testing are shown in Figure 2. The tensile bars tested were through hardened for all materials. The yield strength for the heat treated materials could not be measured and is said to be the same as the ultimate tensile strength. The tensile strength of the new alloy was higher than the FD-0405 even though the alloying content of the material is much less. Both of the diffusion alloyed materials show higher tensile strength compared with the pre-alloyed molybdenum materials. The elongation of the materials was less than 1% in all cases.

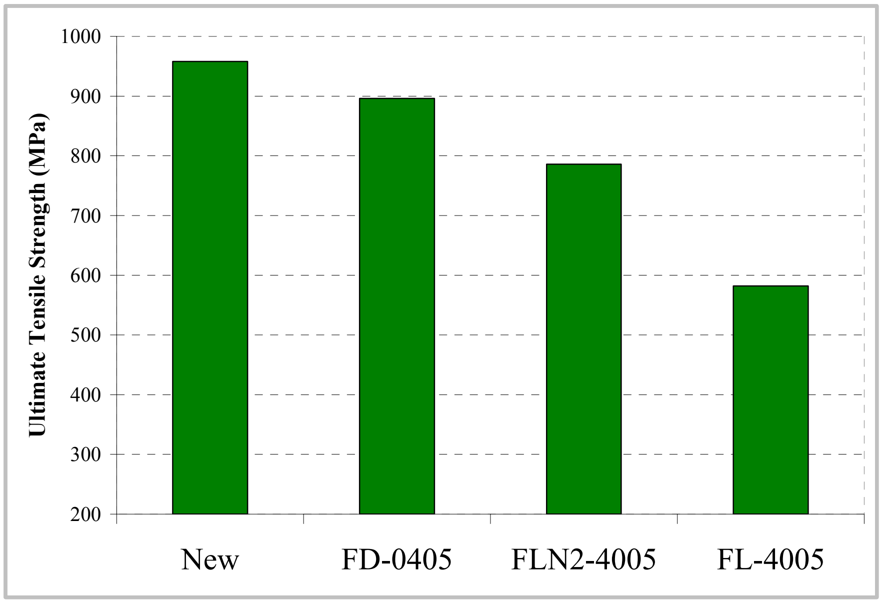

Figure 2. Heat treated ultimate tensile strength of tested materials.

The charpy impact energy of the mixes is presented in Figure 3. The nickel containing materials outperform the non-nickel mix. This is expected as nickel rich areas greatly improve the toughness of PM materials especially in the heat treated condition. The new alloy performs equally to the FLN2-4005 even though the nickel level has been drastically reduced (0.5% vs. 2%). As the FD-0405 has the largest amount of nickel, the impact of this mix is the greatest.

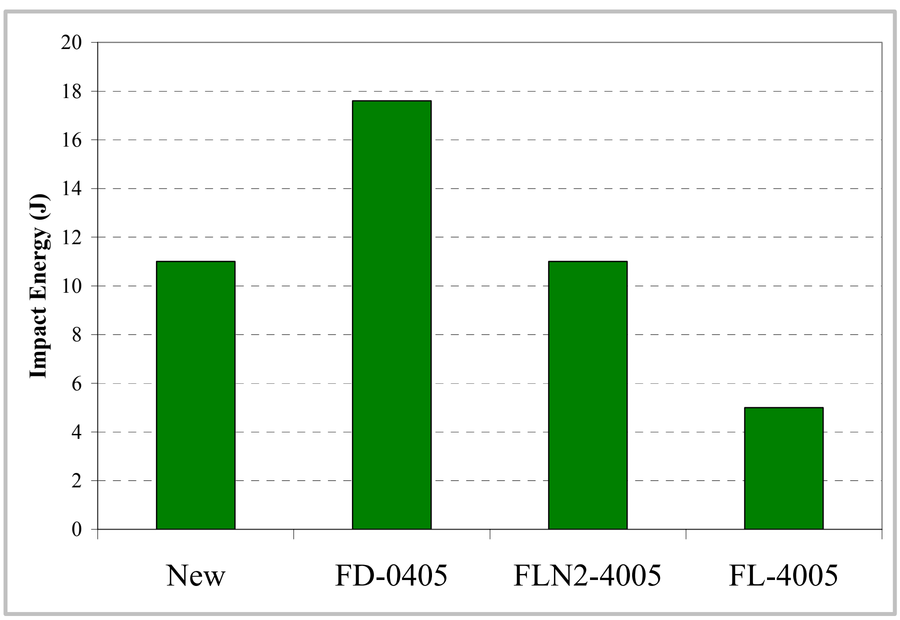

Figure 3. Heat treated impact energy of tested materials.

The transverse rupture strength of the materials follows a similar trend to the ultimate tensile strength. Both of the diffusion alloyed mixes produced higher TRS compared to the pre-alloyed mixes. The new alloy was slightly higher than the FD-0405 in TRS strength. The results are shown in Figure 4.

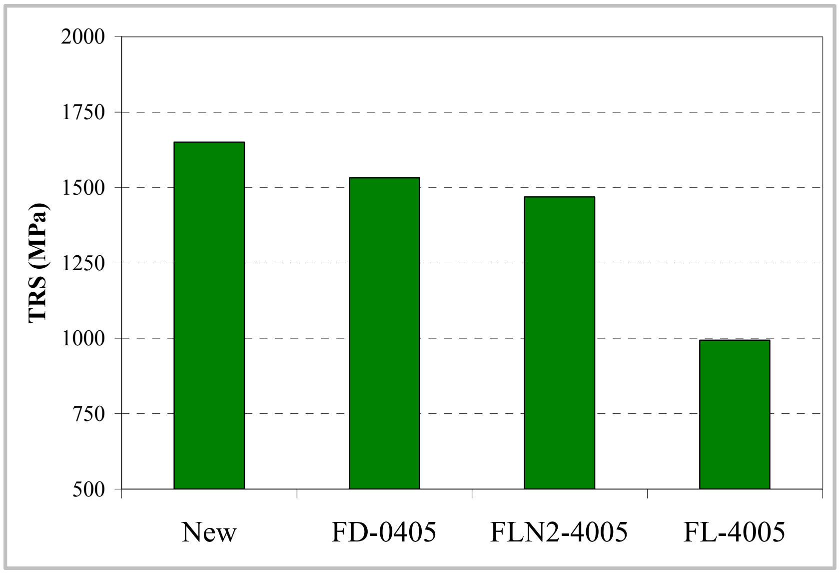

Figure 4. Heat Treated TRS of tested materials.

The apparent hardness results as sintered and as heat treated are shown in Figure 5. All of the heat treated materials had similar hardness. The hardness of the FD-0405 was lower, due to the high amount of nickel which resulted in large nickel rich austenitic areas. A large increase from sintered to as heat treated hardness was observed for the new alloy. The new alloy had the lowest apparent hardness as sintered but the highest as heat treated hardness. The low apparent hardness as sintered has benefits which will be discussed in a later section.

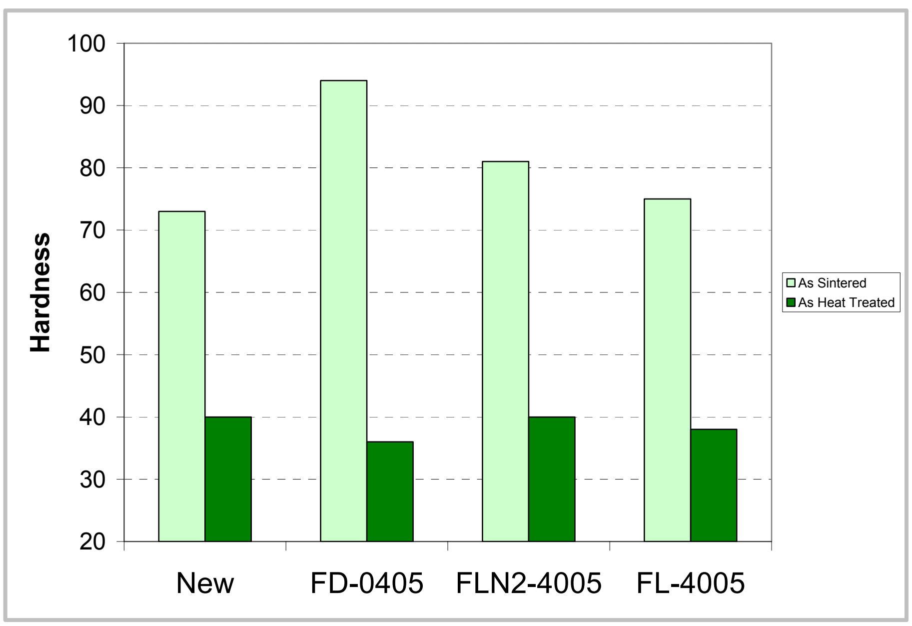

Figure 5. Apparent hardness results as sintered (HRB) and as heat treated (HRC).

Hardenability & Mass Effect

The results of the Jominey end-quench testing are shown in Figure 6.

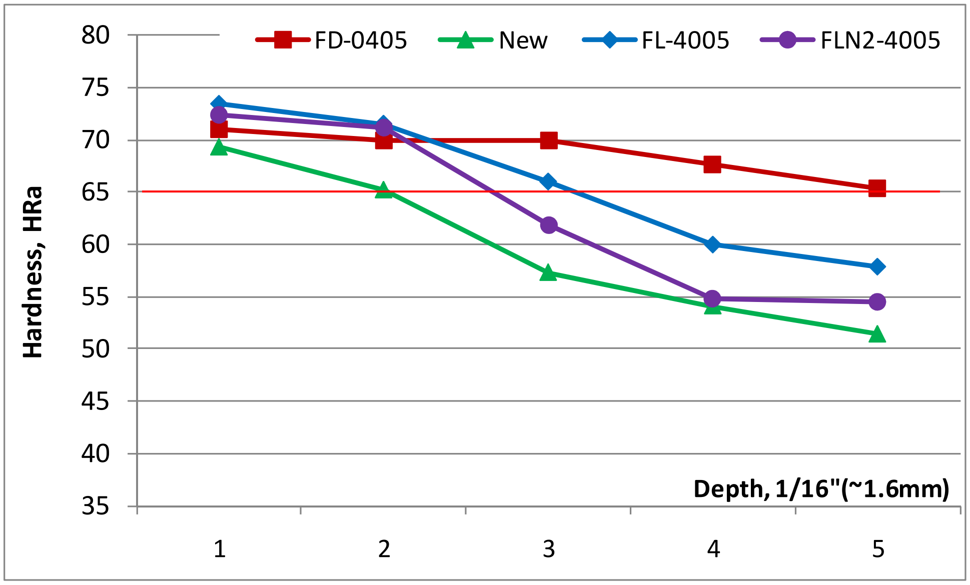

Figure 6. Jominey end-quench results

The most heavily alloyed material, FD-0405, had the highest hardenability. The new diffusion alloyed material showed good hardenability at a J-depth of 2 while the pre-alloyed molybdenum materials had a J-depth of about 3.

Mass effect examinations were conducted on two different size specimens. The results of the 25 mm diameter slug are shown in Figure 7 and the 50 mm diameter slug in Figure 8.

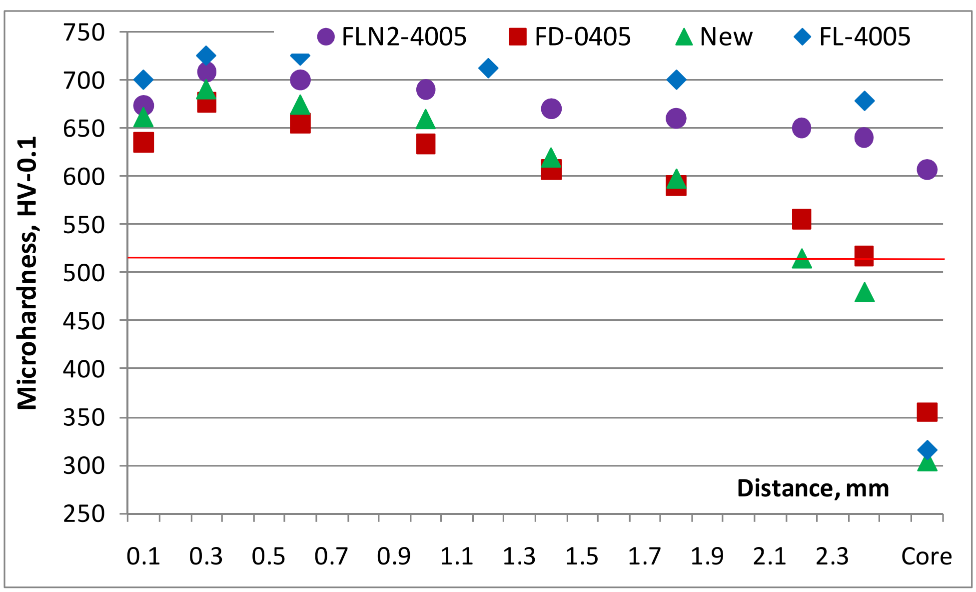

Figure 7. Microindentation hardness profile – 25 mm puck as heat treated.

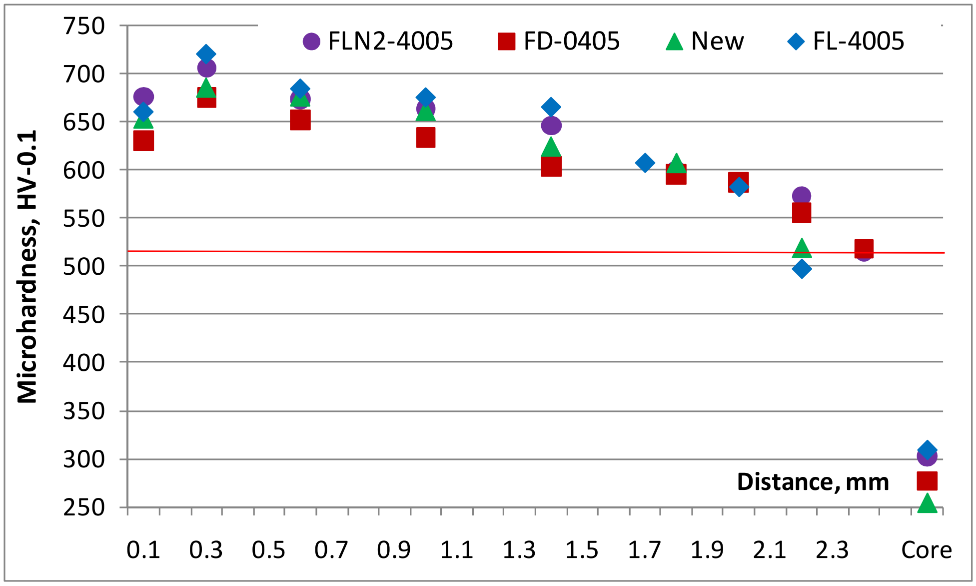

Figure 8. Microindentation hardness profile – 50 mm puck as heat treated.

In the 25 mm slug, both diffusion alloyed materials showed similar behavior. The FL-4005 material was able to form a deeper case compared to the diffusion alloyed materials. The FLN2-4005 slug was through hardened. When moving to the 50 mm slug, all of the materials showed similar behavior and case depth formation. For this size slug, the microindentation hardness values were similar at the core.

Dimensional Stability Study

The standard deviation for out of roundness (OOR) in the as sintered condition is shown in Figure 9. The diffusion alloyed materials show more consistency than the pre-alloyed materials. When heat treated, the standard deviation for OOR almost doubles for each material (Figure 10). Again the diffusion alloyed materials have the lowest standard deviation. The pre-alloyed materials had the highest standard deviation.

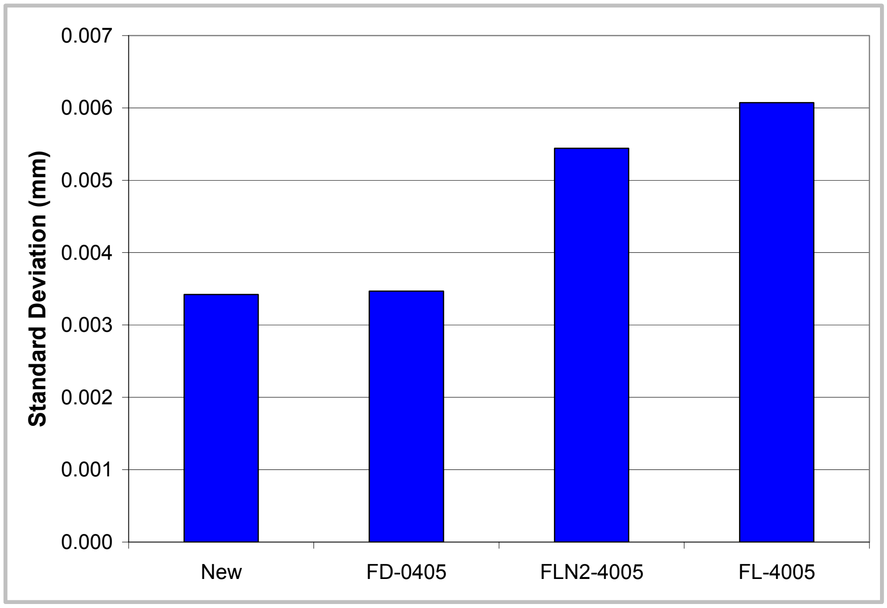

Figure 9. Standard deviation out of roundness – as sintered

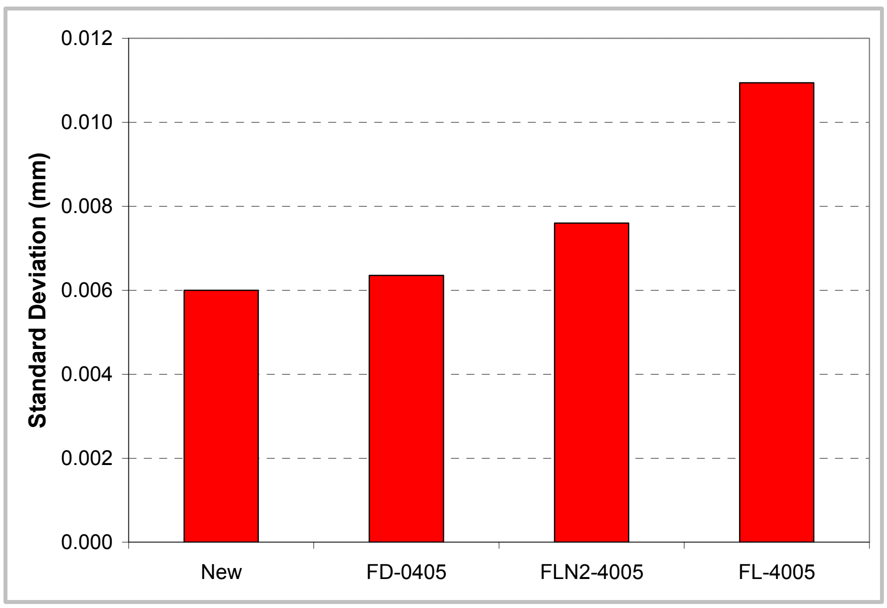

Figure 10. Standard deviation out of roundness – as heat treated

Metallography

The as sintered microstructures are shown in Figure 11. The microstructure of the new alloy was a mixture of ferrite, pearlite, and a small amount of bainite and nickel rich austenite. The FD-0405 material contained a mixture of ferrite, pearlite, nickel rich austenite, bainite and martensite typical the material system. The FLN2-4005 contained a mixture of bainite, dense pearlite and nickel rich austenite. The FL-4005 was almost entirely bainitic except for a small amount of dense pearlite.

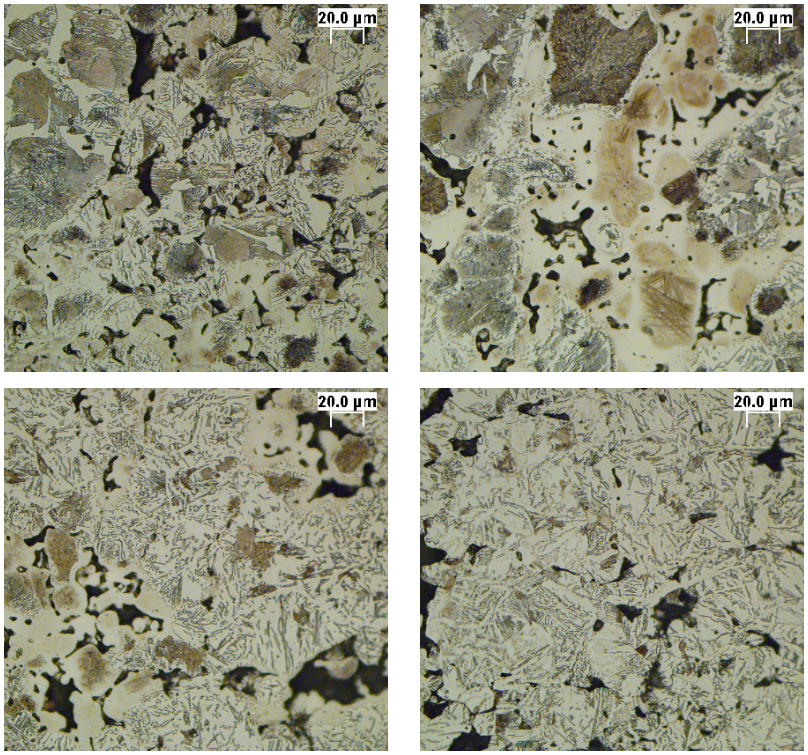

Figure 11. As sintered microstructures (Top : New & FD-0405, Bottom: FLN2-4005, FL-4005).

The heat treated microstructures are shown in Figure 12 and 13. The microstructures presented are from the 25 mm slugs. Similar microstructures were observed in the 50 mm slug.

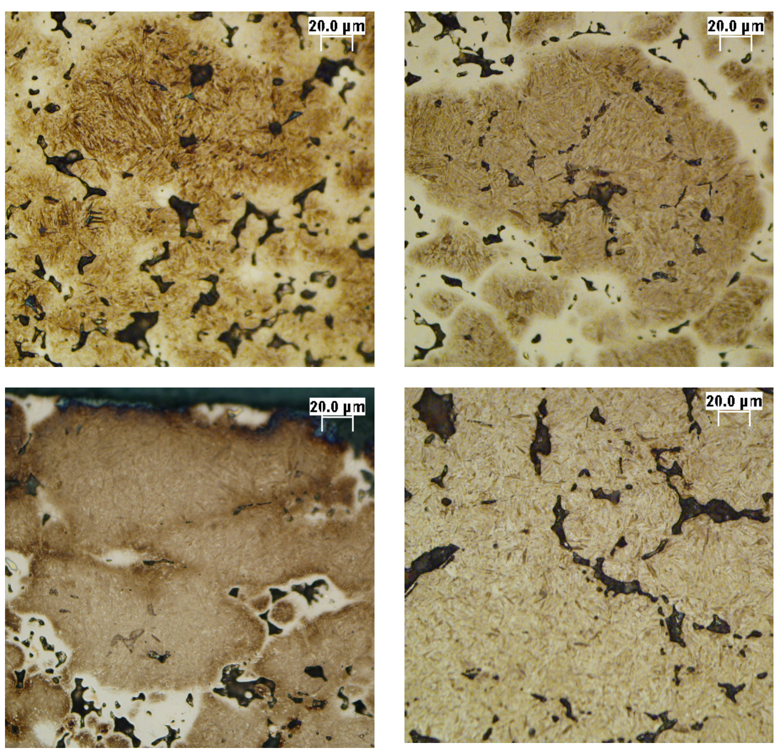

Figure 12. As Heat Treated Microstructures – Surface 25mm Slug
(Top Row: New & FD-0405, Bottom Row: FLN2-4005 & FL-4005)

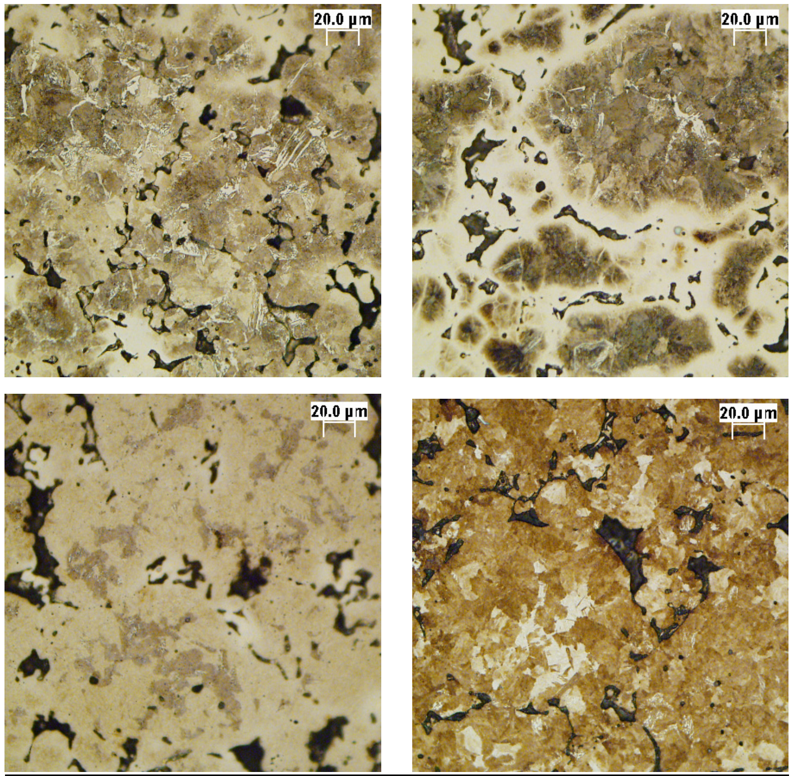

Figure 13. As Heat Treated Microstructures – Center 25 mm slug
(Top Row: New & FD-0405, Bottom Row: FLN2-4005 & FL-4005)

Martensite was observed at the surface for all materials. The nickel containing materials also contained nickel rich austenite regions. The amount of nickel rich austenite present was directly related to the amount of nickel in the mix. The new alloy contained the least amount of nickel rich austenitic areas.

In the center of the slugs, a variety of different microstructures were observed. For the new alloy, a mixture of martensite, bainite, pearlite, and nickel rich austenite was found. A very small amount of ferrite was also observed in certain regions. For the FD-0405 material, the center contained the same phases as the new alloy, although the amount of martensite was greater. This is expected due to the difference in alloying between the two materials. The FLN2-4005 material contained a mixture of martensite, bainite and nickel rich austenite. The FL-4005 material contained a mixture of pearlite, ferrite and a small amount of bainite.

DISCUSSION

In the as sintered condition, the new alloy shows medium level strength and hardness levels. The microstructure is mostly pearlite and ferrite compared to the FD-0405 which is a mixture of all phases and primarily bainitic for the pre-alloyed molybdenum based materials. Because of the lower strength phases, the machinability and workability in the as sintered condition are expected to be much better than the other material systems. The stability of the alloy in the heat treated condition could allow for more machining to be done in the as sintered condition. In the heat treated condition, large increase in mechanical properties was observed for the new alloy. Tensile strengths and apparent hardness levels greater than the heavily alloyed FD-0405 material system were achieved. The level of dimensional stability in the heat treated condition for the new alloy outperformed the other material systems tested. The combination of strength and stability make the new alloy a possibility to replace more heavily alloyed material systems in high performance applications.

As the test pieces were through hardened, caution must be taken in the heat treatment process. The hardenability and mass effect study show the new alloy to be less hardenable than the other alloys. Optimization of the heat treatment parameters will be necessary when converting to the new alloy. The impact energy of the new alloy was less than the FD-0405 but similar to the FLN2-4005. For impact critical components, the effect of the lower nickel content should be considered.

CONCLUSIONS

The following conclusions can be drawn from this study:

- The new lean diffusion alloy, D.AQ, showed medium tensile strength and hardness levels in the as sintered condition. When heat treated, the alloy was able to out perform the more heavily alloyed FD-0405 material system with regards to tensile strength and hardness.
- The as sintered hardness and microstructure should allow the new alloy to have better machinability and workability than the other material systems. This should allow the alloy to be more robust in the manufacturing process.
- By using lower alloying levels and the diffusion alloying method, the new alloy achieved better dimensional consistency after heat treatment compared to the other alloys.
- The hardenability of the new alloy was less than the more heavily alloyed systems. Optimization of the heat treatment parameters should be conducted to achieve a through hardened component.

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