Martensitic PM Materials

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ABSTRACT

Components often require high strength, high hardness, or high wear resistance to meet the demands of high performance applications. To meet these requirements, a martensitic microstructure is required. In powder metallurgy (PM), the martensitic structure can be achieved through conventional heat treatment or through sinter hardening. PM offers unique alloying techniques which can be used to develop the desired microstructure. Even if the resulting microstructure is predominantly martensitic, differences in final performance are often observed. In this paper, common alloying systems are compared in the martensitic condition. The microstructure and resulting properties of the different alloys are compared and discussed.

INTRODUCTION

Heat treatment is a common method applied to ferrous materials to improve the strength and wear resistance. Desired changes occur in both the properties and the structure of a part when a heat treatment is used. During the process, a martensitic microstructure is formed [1]. One of the most widely used methods of obtaining a martensitic microstructure is through neutral hardening, also known as quench and tempering.

After sintering, the components are heated to an austentizing temperature ranging from 815 to 870 ˚C (1500 to 1600 ˚F). They are held under a carbon potential similar to the carbon level in the base material. After a desired period of time, the components are rapidly cooled using quenching techniques. The quenching causes the martensitic microstructure to form. Quenching or rapidly cooling process creates stress in the component. The stress causes the components to become brittle which makes tempering a necessary step in the heat treat process [2].

Tempering is the process in which the components are heated to relieve stresses, complete the martensite transformation, and to ensure dimensional consistency. Tempering is typically completed in the temperature range of 150 to 595 ˚C (302 to 1103 ˚F) and for various times. This final step in the heat treating process increases ductility and adds toughness to the material [2].

In addition to heat treatment, various alloying elements can be incorporated in a ferrous material to enhance the properties. Different methods can be used to achieve the desired properties. Common types of alloying systems in PM can be used for a heat-treated material include elemental, diffusion-alloyed, and pre-alloyed. The benefit of using different alloying elements and techniques allows the flexibility to customize the material to fit the component requirements [3]. Each of the alloying elements provides a certain hardenability which leads to improved properties. Copper, nickel, and molybdenum are a few of the alloying elements used in PM.

Admixing copper can significantly improve the properties of a material. Copper has a melting point of 1083 ˚C (1981 ˚F) which is lower than conventional sintering temperatures. Since the melting point is lower, the copper melts and diffuses during sintering. The grain boundaries are strengthened and the particle bonding is improved. This process results in swelling within the component [4, 5]. To help control the dimensions, copper is used in combination with carbon. During sintering, the copper can significantly improve the strength characteristics but it does not provide the same benefit in the heat treat condition [6, 7].

When nickel is used in a material, areas of nickel-rich austenite will form. The melting point of nickel is 1455 ˚C (2651 ˚F) which is higher compared to conventional sintering temperatures. The nickel partially to diffuses to the iron during sintering. During cooling, the nickel rich austenite areas stabilize. The stabilized austenite improves ductility, which in turn improves the strength of the material [7, 8].

Molybdenum is used in applications where high hardenability is needed. The hardness and strength properties can be improved by just by adding molybdenum to the material, but it can provide even more of an improvement when it is used in combination with nickel [2]. Typically, molybdenum is used in material systems where the alloying content is added during the manufacturing process of the powder.

The alloying elements commonly found in diffusion-alloyed materials are nickel, copper, and molybdenum. During the manufacturing process the alloying elements are bonded to the base iron in a secondary annealing step. Typically diffusion-alloyed materials are used when high wear resistance and high strength is needed. By diffusing the alloying elements to the base iron, this can improve the strength and hardenability of the material while eliminating segregation of the alloy elements. [9]

In pre-alloyed materials, the alloying elements are added during the melt prior to atomization. Thus, results in a uniform and homogenous structure in the powder particle. Typically pre-alloyed materials are used when high wear resistance and high strength is needed [9]. Common elements pre-alloyed include molybdenum, nickel, manganese, and chrome.

In this study, common alloying systems were compared in the martensitic condition. Test specimens were prepared to evaluate the hardness and strength of various materials within each system. A gear trial was completed to further evaluate the strength, microstructures, and fracture surfaces of the materials and to compare and contrast each alloying system.

EXPERIMENTAL PROCEDURE

Materials

The alloying systems used for this study were elemental, diffusion-alloyed, and pre-alloyed. The materials were chosen based on the type of base irons that are commonly used for each alloying system. The base iron and alloy percentages are shown in Table 1.

Table 1: Base Iron Compositions

Description	Base	Cu	Ni	Mo
Iron	ASC100.29	-	-	-
Diffusion-alloyed	D.AB	1.50	1.75	0.5
	D.AE	1.50	4.0	0.5
Pre-alloyed	Astaloy® 85 Mo	-	-	0.85

The base irons were used to manufacture the mixes shown in Table 2. Each mix is represented by the MPIF designation codes.

Table 2: Mix Compositions

Base	Cu	Ni	Graphite	Lubricant	MPIF Code
ASC100.29	2%	-	0.85%	0.75% Amide Wax	FC-0208HT
ASC100.29	-	2%	0.85%	0.75% Amide Wax	FN-0208HT
D.AB	-	-	0.55%	0.75% Amide Wax	FD-0205HT
D.AE	-	-	0.55%	0.75% Amide Wax	FD-0405HT
Astaloy® 85 Mo	-	-	0.55%	0.75% Amide Wax	FL-4405HT
Astaloy® 85 Mo	-	2%	0.55%	0.75% Amide Wax	FLN2-4405HT

Heat Treat Properties

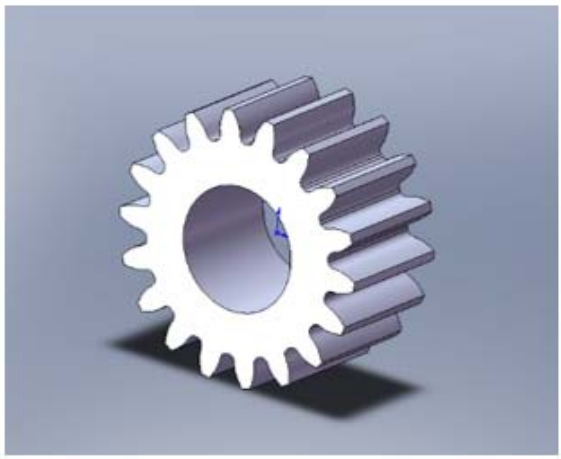
Two types of test specimens were prepared to evaluate the heat treat properties of each material. Five transverse rupture strength (TRS) bars and five impact (IE) bars were compacted to achieve a sintered density of 6.9 g/cm^3. All bars were sintered in a laboratory belt furnace at 1121 °C (2050 °F) for 20 minutes in a 90/10 N_2/H_2 atmosphere with a normal cooling rate of 0.5 °C/sec (1 °F/sec).

After sintering, the bars were sent to Advanced Heat Treating, Inc. (St. Mary's Pennsylvania) for neutral hardening. The bars were austenitized at 843 ˚C (1550 ˚F) for 1 hour. The carbon potential for the FC-0208HT and FN-0208HT materials was 0.8 % C. The carbon potential for all other materials was 0.6 % C. The specimens were then oil quenched and tempered at 204 ˚C (400 ˚F) for 1 hour.

Upon completion of the neutral hardening, the bars were evaluated for density, carbon content, apparent hardness and TRS. The IE bars were evaluated for impact energy. The testing was completed in accordance with MPIF standards [9].

Gear Trials

Ten gears were compacted from each material to achieve a green density of 6.9 g/cm^3. The gears were sintered and neutral hardened following the same conditions as the TRS and IE bars. The gear parameters are shown in Figure 1.



HB Gear	
No. of Teeth	18
Module	1.56
Pressure Angle	20 °
Major Dia.	31.5 mm
Minor Dia.	24.5 mm
Root Radius	0.69 mm
Tooth Thickness	2.4 mm

Figure 1: Gear parameters

Two gears from each material were evaluated for density, carbon content, and apparent hardness. Eight gears from each material were evaluated for gear tooth break load.

Upon completion of the heat treat properties and the gear trial, microstructures and fracture surfaces were analyzed to compare and contrast the materials and the alloying systems.

RESULTS

Heat Treat Properties

The density, carbon content, apparent hardness, and impact energy for each material is presented in Table 3.

Table 3: Heat Treat Properties

Mix ID	Density (g/cm^3)	Carbon (%)	HRC	IE (J)
FC-0208HT	6.90	0.79	38	7
FN-0208HT	6.92	0.78	39	6
FD-0205HT	6.93	0.61	37	6
FD-0405HT	6.91	0.62	38	7
FL-4405HT	6.90	0.62	36	4
FLN2-4405HT	6.92	0.60	37	5

A density of 6.9g/cm^3 was obtained for each material. The carbon content for the FC-0208HT and FN-0208HT materials was 0.8%. The carbon content for all other materials was 0.6%. All of the materials had a similar apparent hardness. The impact energy was slightly lower for the pre-alloyed materials compared to the elemental and diffusion-alloyed materials. The TRS results are shown in Figure 2.

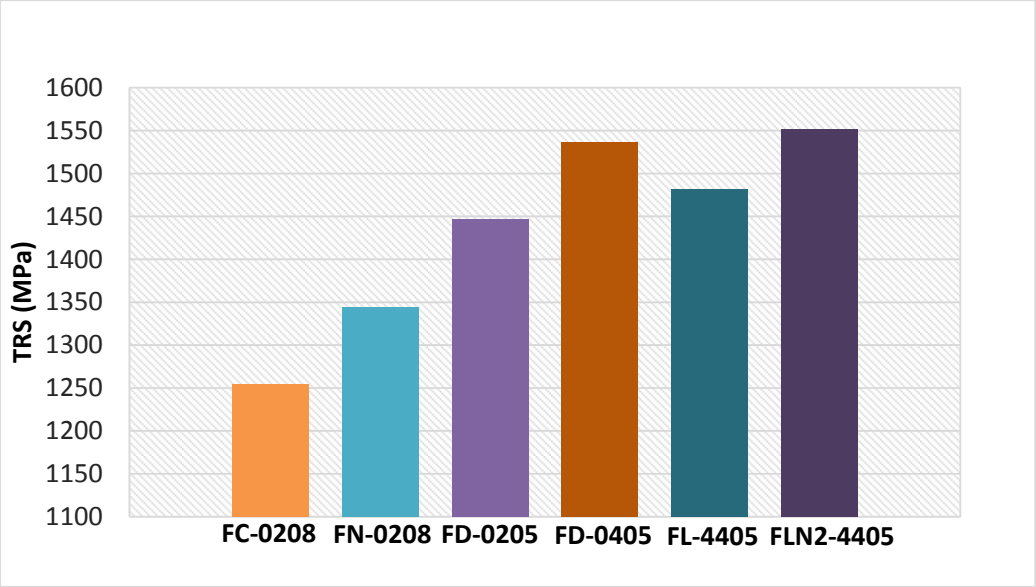


Figure 2: TRS comparison of the elemental, diffusion-alloyed, and pre-alloyed materials

The elemental materials had a lower TRS compared to diffusion-alloyed and pre-alloyed materials. The FLN2-4405HT material had the highest strength compared to the other materials.

Gear Trial

The gears were tested for density, carbon content, apparent hardness, and strength. The density, carbon content, and apparent hardness results are shown in Table 4.

Table 4: Gear Properties

Mix ID	Density (g/cm^3)	Carbon (%)	HRC
FC-0208HT	6.93	0.78	38
FN-0208HT	6.91	0.79	41
FD-0205HT	6.90	0.61	39
FD-0405HT	6.91	0.60	38
FL-4405HT	6.93	0.62	41
FLN2-4405HT	6.95	0.62	40

A density of 6.9 g/cm^3 was obtained for each of the materials. The carbon content was similar between each of the materials. The carbon content for the FC-0208HT and FN-0208HT materials was 0.8%. The carbon content for all other materials was 0.6%. The apparent hardness was similar to the apparent hardness of the TRS bars. The gear tooth break load values are shown in Figure 3.

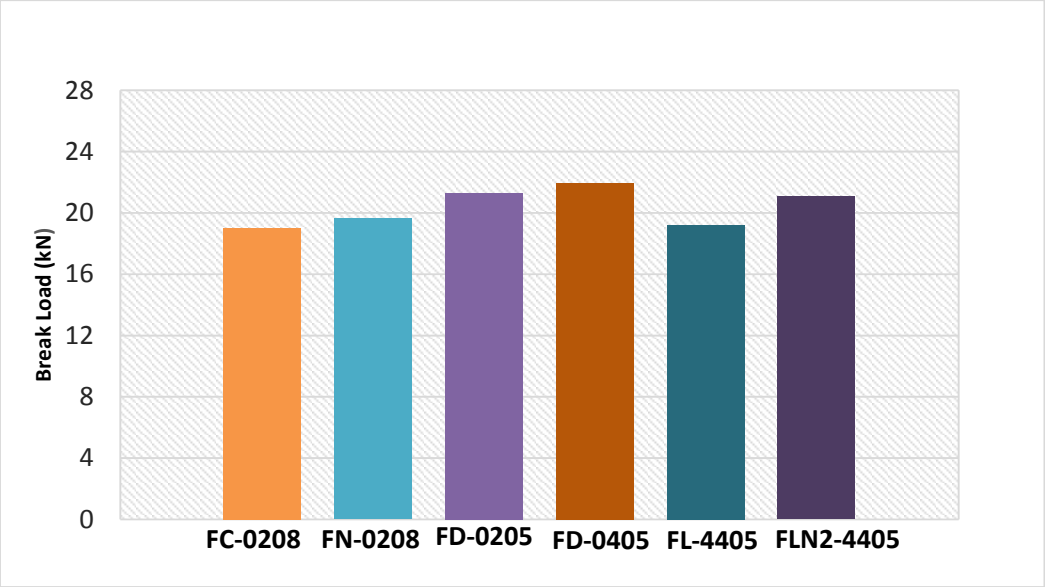


Figure 3: Gear tooth break load comparison of the elemental, diffusion-alloyed, and pre-alloyed materials

The FC-0208HT and FN-0208HT materials had a similar breaking load. The FD-0405HT material had the highest strength compared to the other materials.

Microstructures

Microstructure analysis was completed on the gears to understand how each of the systems relate to one another. The heat treatment resulted in a martensitic microstructure. The microstructures for each material are shown in Figures 4-9.

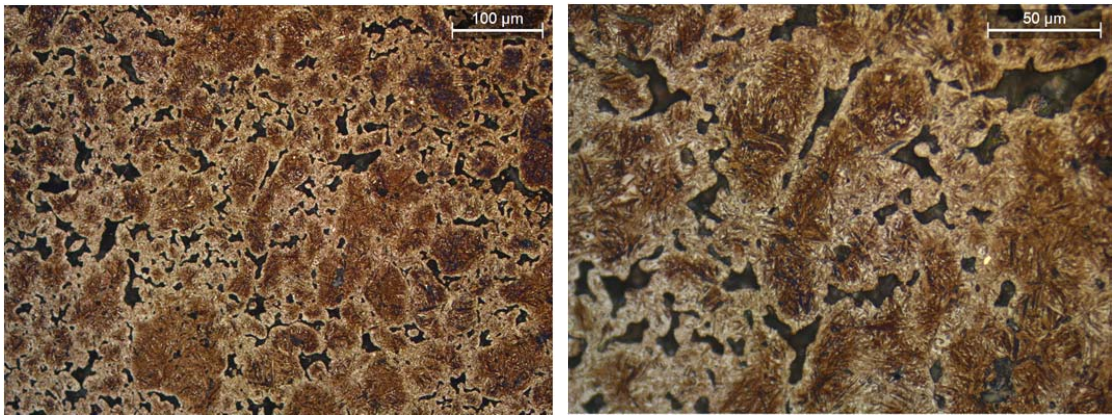


Figure 4: FC-0208HT

In the FC-0208HT material, high amounts of retained austenite were seen at the particle boundaries.

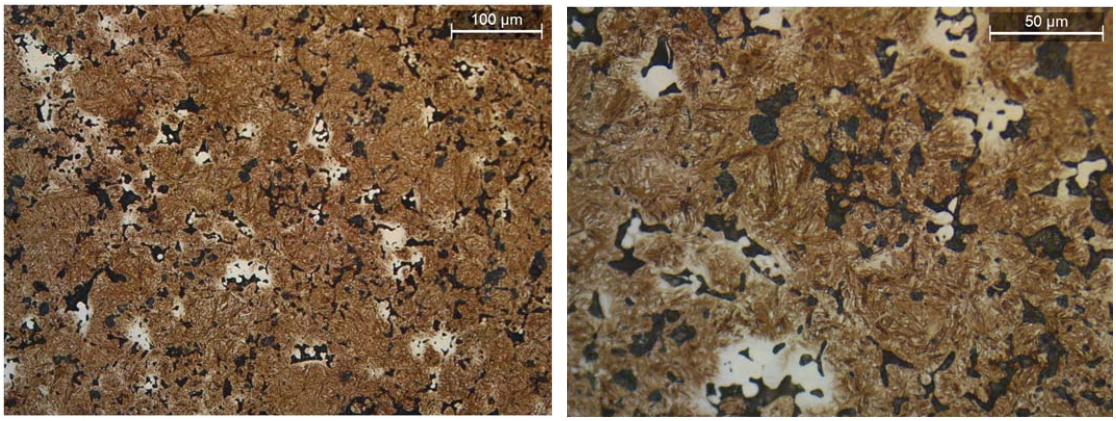


Figure 5: FN-0208HT

Compared to the FC-0208HT material, the areas of martensite in the FN-0208HT material were uniform throughout the structure. The elemental nickel resulted in the formation of nickel-rich austenite.

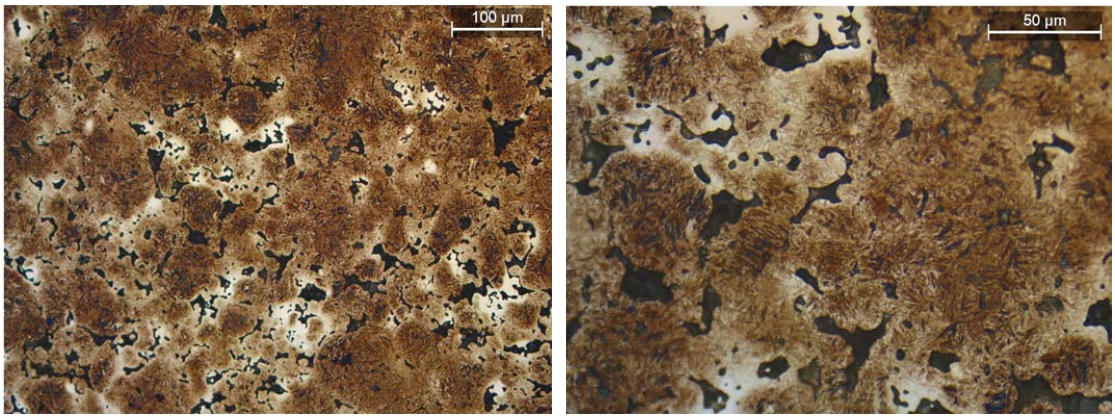


Figure 6: FD-0205HT

The microstructure of the FD-0205HT gear was martensitic with areas of nickel-rich austenite. The structure was similar to FN-0208HT.

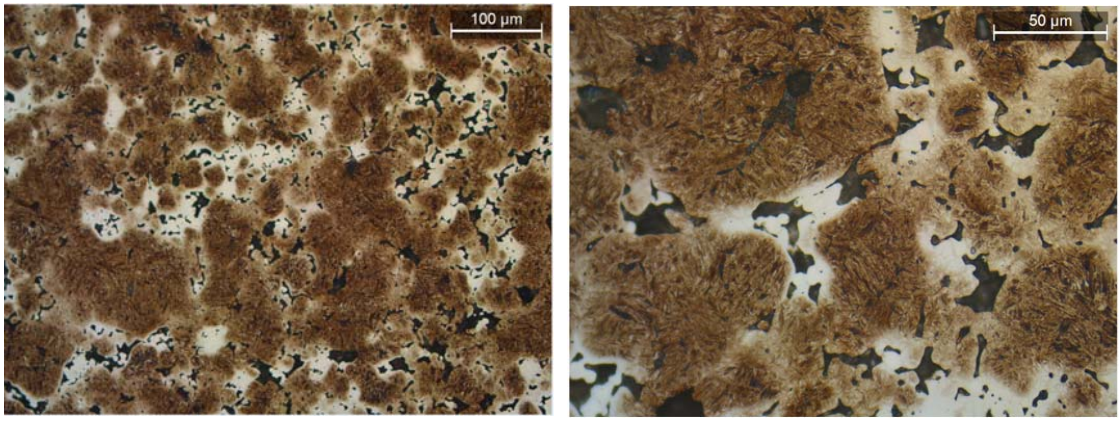


Figure 7: FD-0405HT

The microstructure of the FD-0405HT gear was martensitic with areas of nickel-rich austenite. There was an increase in the percentage of nickel-rich austenite for the FD-0405HT material compared to the FD-0205HT material due to the additional nickel added to the alloy.

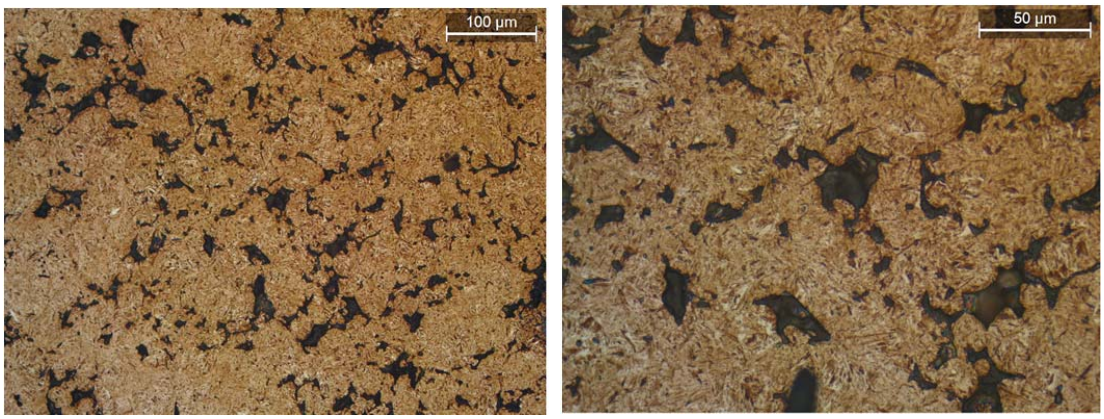


Figure 8: FL-4405HT

Compared to the elemental and diffusion-alloyed materials, the microstructure of the FL-4405HT was homogenous throughout the gear.

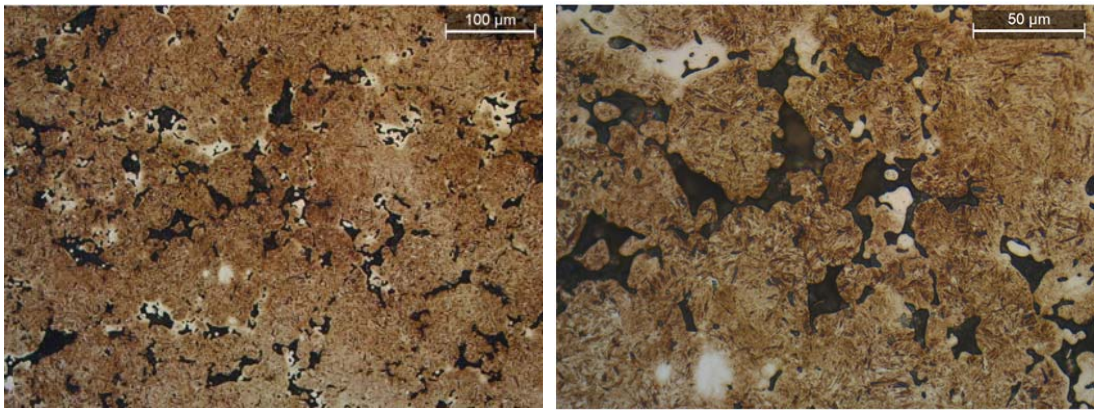


Figure 9: FLN2-4405HT

The microstructure of the FLN2-4405HT gear was similar to the FN-0208HT material with martensite and areas of nickel-rich austenite.

The microhardness for each material is shown in Table 5.

Table 5: Microhardness

MPIF Code	$MHV_{0.1}$
FC-0208HT	710 (654-766)
FN-0208HT	721 (680-787)
FD-0205HT	740 (735-745)
FD-0405HT	745 (735-755)
FL-4405HT	732 (716-755)
FLN2-4405HT	733 (716-745)

A similar average microhardness was observed between each of the material systems. The elemental materials had a slightly lower microhardness and a wider range compared to the diffusion-alloyed and pre-alloyed materials.

DISCUSSION

As expected, the heat treatment resulted in the formation of martensite for each material. For the TRS bars, lower strength values were seen for the FC-0208HT and FN-0208HT elemental materials. The highest strength was achieved for the FD-0405HT and FLN2-4405HT materials. A further improvement in the strength was seen in the diffusion-alloyed and pre-alloyed materials compared to the elemental materials. The mixes containing nickel showed improvements in strength compared to the mixes without nickel. One major difference was observed between the TRS strength values and the gear tooth break load. The TRS strength varied for each material compared to the gear tooth break load. To further the study, a fracture surface analysis was completed to examine how the nickel effected the strength of the materials.

Fracture Surfaces

The fracture surfaces of the gears and the TRS bars followed a similar trend. The TRS photographs were used for the comparison of the materials. The fracture surfaces and strength for each material are shown in Figures 10-15.

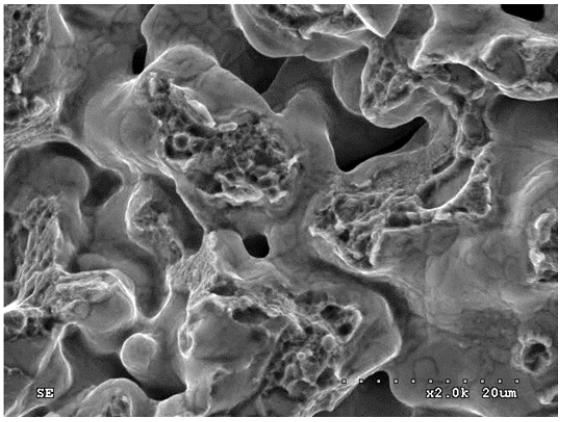


Figure 10: FC-0208HT – 1254 MPa

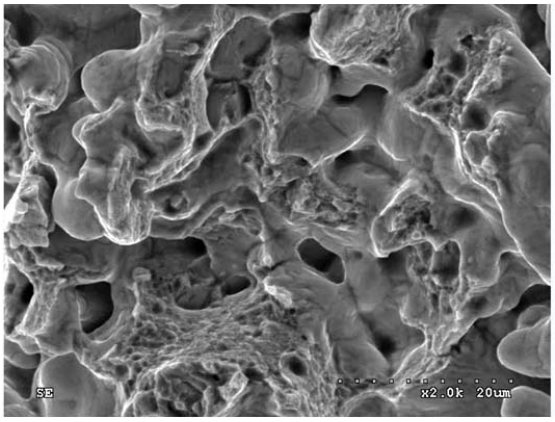


Figure 11: FN-0208HT – 1344 MPa

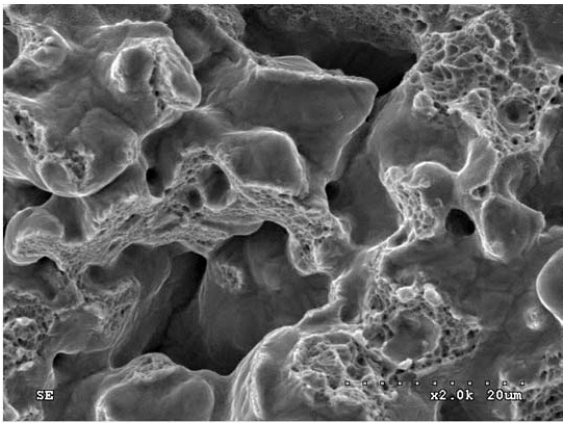


Figure 12: FD-0205HT – 1447 MPa

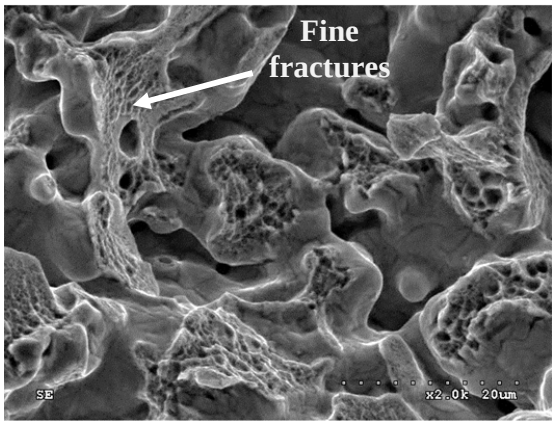


Figure 13: FD-0405HT – 1537 MPa

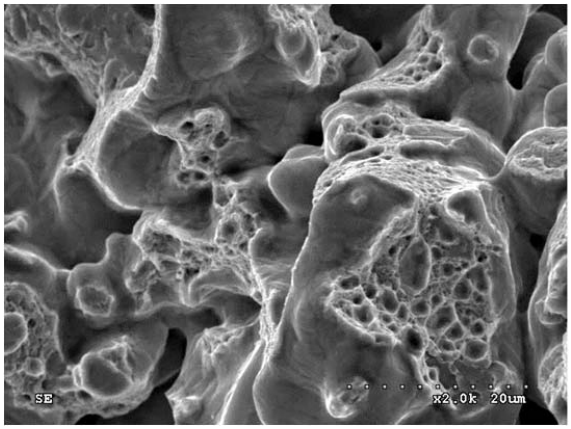


Figure 14: FL-4405HT – 1482 MPa

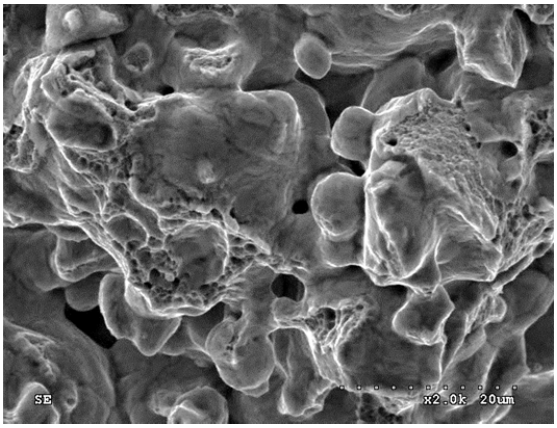


Figure 15: FLN2-4405HT – 1551 MPa

The fractures surfaces exhibit cup-cone fractures and fine fractures. The cup-cone fracture has dimpling at the surface. The elemental materials showed less dimpling compared to the diffusion-alloyed and pre-alloyed materials. Fine fractures were observed in the mixes containing nickel.

The strength is related to the features shown in the fracture surfaces. The elemental materials had lower strength values and less dimpling at the surface. The diffusion-alloyed and pre-alloyed materials had increase in strength and showed more dimpling at the surface. Fine fractures were seen in the mixes containing nickel which had higher strength values.

A comparison of the microstructures and fracture surfaces for the FL-4405HT and FLN2-4405HT materials are shown in Figures 16-19.

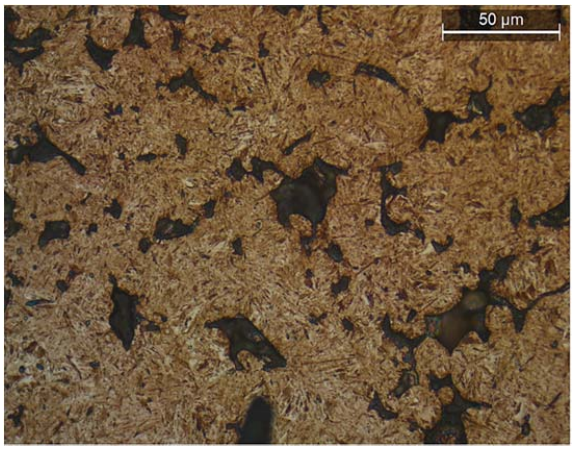


Figure 16: FL-4405HT

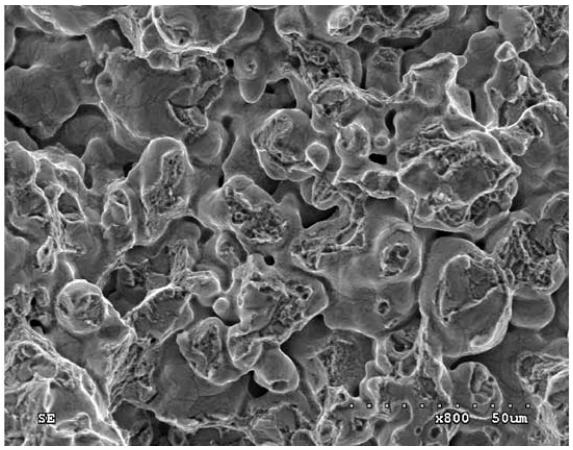


Figure 17: FL-4405HT

The FL-4405HT material had a fully martensitic microstructure. The fracture surface showed dimpling. No fine fractures were observed.

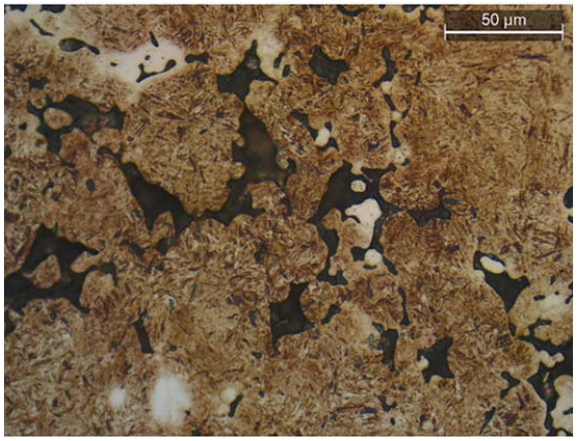


Figure 18: FLN2-4405HT

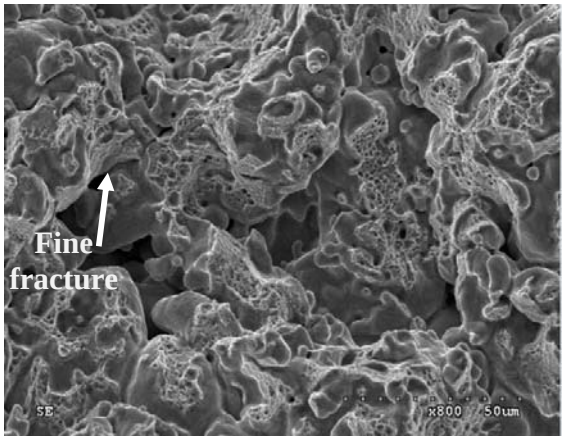


Figure 19: FLN2-4405HT

When nickel is added to a material, areas of nickel-rich austenite are formed. The fine fractures indicate where the areas of nickel-rich austenite were formed in the microstructure.

CONCLUSION

The following conclusions can be drawn from the completion of this study:

- During the heat treatment process, a martensitic microstructure is formed which improves the strength characteristics of a material.

- The TRS strength varied between each of the materials compared to the gear tooth break load. The gear tooth break load was similar between each of the materials.

- Nickel improves the strength of the material. Nickel results in areas of nickel rich austenite which stabilizes during the heat treatment process. The stabilized nickel rich austenite provides added ductility and an increase in strength.

- In the areas where the nickel rich austenite was formed, fine fractures were observed on the fracture surfaces.

REFERENCES

[1] *Steel Heat Treatment Handbook*/Totten, George E., Howes, Maurice A. Editors (1997) Marcel Dekker Inc. pg. 1

[2] Dossett, J.L., & Boyer, H.E. (2006). Practical Heat Treating Second Edition. ASTM International. pgs. 5-6, 130

[3] Edmond Llia, PMT; George Lanni; Constance Thompson, PMT; Kevin T. Tutton; "Another Look at the Effect of Copper on the Mechanical Properties of As-Sintered Iron-Copper-Carbon-Alloys". International Journal of Powder Metallurgy, 2013 pp. 35-42.

[4] Michael M. Marucci; Francis. Hanejko. "Effect of Copper Alloy Addition Method on the Dimensional Response of Sintered Fe-Cu-C Steels." MPIF-Advances in Powder Metallurgy and Particulate, 2010, Part 7. pp 11-20.

[5] S. Ashok; N. Ramesh; A.X. Kennedy. "Dimensional Properties of the Mix FC-0208 with Different Copper Addition." MPIF Advances in Powder Metallurgy and Particulate. 2001, Part 5, pp. 312-319.

[6] Singh, T., Stephenson, T., & Campbell, S. (n.d.). "Nickel-Copper Interactions in P/M Steels".

[7] Amber Neilan; Sarah Ropar; Bo Hu; R. Warzel III. "Characterization of Nickel Additions to Heat Treated Iron-Copper-Carbon Materials." *Advances in Powder Metallurgy & Particulate Materials*, complied by I.E. Anderson and T. Pelletiers, Metal Powder Industries Federation, Princeton, NJ, 2014, Part 7, pp. 15-24.

[8] *Steel Heat Treatment: Metallurgy and Technologies*/Totten, George E., Howes, Maurice A. Editors (2007) CRC Press Taylor & Francis Group (pg. 760)

[9] *MPIF Standard 35 Material Standards for PM Structural Parts.* (n.d), MPIF

[10] Thomas F. Murphy; Michael C. Baran. "An Investigation into the Effect of Copper and Graphite Additions to Sinter-Hardening Steels." *Advances in Powder Metallurgy and Particulate*, 2004, Part 10, pp. 266-274.