

# **High Nickel Alloys Offering a Combination of High Strength and High Impact Properties**

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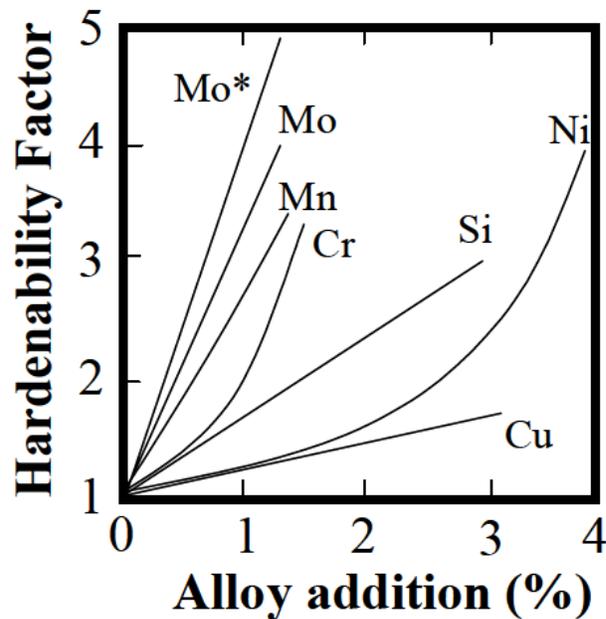
## **ABSTRACT**

High nickel steels offer the opportunity to produce powder metallurgy components with excellent high strength and ductility combinations. These alloys are a sinter-hardenable class of materials that do not require conventional heat treatment processes to develop these unique properties. The alloys must be sintered at high temperatures in order to obtain the value of the elemental additives that are included in these ferrous alloys - as well as the enhancement of the densification process. As-sintered properties include UTS values near 1380 MPa (200 ksi), yield strengths over 827 MPa (120 ksi) and elongations over 2%. In addition, impact energy values are over 40 J (30 ft. lbf.). A post-sinter temper/draw cycle did not further improve mechanical properties of the PM steels with higher Ni contents, and was deemed unnecessary. When component failure due to insufficient ductility is not an option, these materials can readily justify the slightly higher cost.

## **INTRODUCTION**

The powder metallurgy (PM) process is an extremely flexible method of component manufacturing that can result in highly ductile (>30% elongation) or very strong (>1500 MPa UTS) products. This wide range of properties is possible because of the relative ease with which compositional changes can be made and the many possible process parameters that can be controlled (compaction pressures, sintering time and temperatures, post-sinter operations, heat treatment, surface modifications, etc.). In support of these objectives to fulfill the wide-ranging needs of customers, powder producers have developed highly engineered powders that offer great consistency in powder purity, morphology, and particle size distribution. Further enhanced value is delivered to PM component makers with alloyed, partially alloyed, and bonded powder combinations.

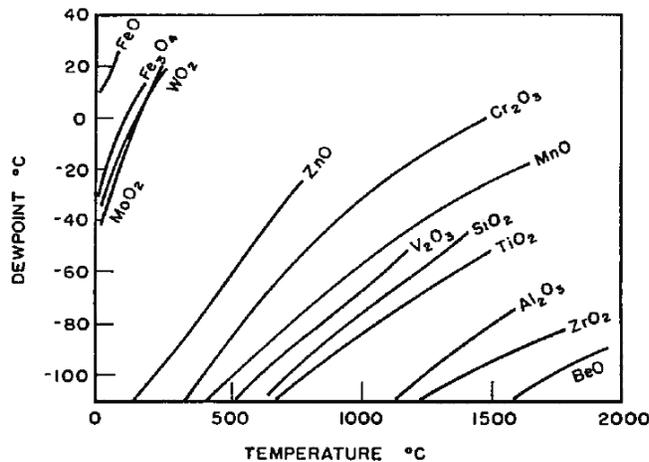
In the specific case of ferrous powder metallurgy, the degree of hardening or strengthening imparted by addition of some selected metals is shown <sup>1,2</sup> in Figure 1. Perhaps the most important of these metals is Mo, since a relatively small amount is needed to create significant hardenability. The element is not detrimental to the compressibility of the base Fe during molding<sup>3</sup> and, furthermore, is a ferrite stabilizer, which enhances the sintering process due to the higher diffusion rates in the more open b.c.c. structure. For more than two decades, the PM industry has utilized Mo because of its ability to harden iron, and is the basis of many pre-alloyed powders intended for high strength structural parts.



**Figure 1.** Hardenability factors for selected pre-alloyed elements in Fe.<sup>1</sup> Mo\* is the hardenability factor of the element for Ni contents >0.75 wt.%.

McQuaig and Sokolowski<sup>4</sup> confirmed that low levels (0.3% to 0.5%) of pre-alloyed Mo are sufficient to cause hardenability in parts with thin cross sections, but greater levels (0.85% or higher) are needed for large cross-section parts. Furthermore, they also showed that admixed Ni was better for end-product mechanical properties than pre-alloyed Ni, because the latter route decreased compressibility at the molding stage. A study of dynamic properties<sup>5</sup> concluded that increasing levels of Mo, improved fatigue strength, but beyond 0.85 %Mo, no additional benefit was gained. The improved fatigue properties from of 0.5 % to 0.85 %Mo were thought to be due to the change in microstructure from a divorced pearlite/pearlite type to a bainite/martensite type. Again, affirming the role of Mo in enhancing hardenability of the steel, as the level of this alloying element is increased.

Additional strengthening is achieved by alloying with Cu, Si, Cr, Mn and Ni, as shown in Figure 1. Mn, however, has the disadvantage of decreasing compressibility<sup>3</sup> and, to make matters worse, has a high affinity for oxygen.<sup>6</sup> Along with Mn, the stable oxides of Cr and Si are more difficult to reduce (Figure 2) in conventional sintering temperatures and atmospheres.<sup>2, 6-9</sup>



**Figure 2.** The equilibrium temperature for reduction of selected metal oxides as a function of hydrogen dew point; components in assumed to be in pure, standard, states.<sup>6</sup>

However, even with this known attribute of Mn, Cr and Si, several alloys have been commercialized which can successfully produce high performance components as long as care is taken to ensure that dry sintering atmospheres are used.<sup>2, 8-12</sup>

Both Cu and Ni have been used quite extensively in the PM industry as they offer a good combination of structural properties with ease of PM manufacturing, but larger amounts are needed to attain the strengthening levels comparable to that of Mo. On the plus side, Mo and Ni are reported to have a synergistic effect on hardenability.<sup>2, 5, 13-15</sup> This is why there are two curves for Mo in Figure 1. The Mo\* curve is the hardenability factor when Ni in amounts greater than 0.75% is present in the material.

This current study reviews the effect of %Ni, %C, and sintering temperature on the mechanical properties of the Fe-Ni-C alloy system. The base pre-alloyed powder Fe-0.85%Mo is used in all the test blends because it provides a powerful starting material to promote hardenability in the final product.

## **EXPERIMENTAL**

Batches of test blends were made using Fe powder pre-alloyed with 0.85%Mo, and varying levels of Ni and C content. INCO 123 was selected as the source for elemental Ni powder, while 0.75% Acrawax C was selected as the lubricant. Tensile and Impact bars were compacted at various pressure ranging from 414 (30 tsi) to 827 MPa (60tsi). Test samples were sintered at 1121 °C (2050 °F), 1204 °C (2200 °F), and 1288 °C (2350 °F) using a sintering atmosphere that was nominally 67% hydrogen and 33% nitrogen.

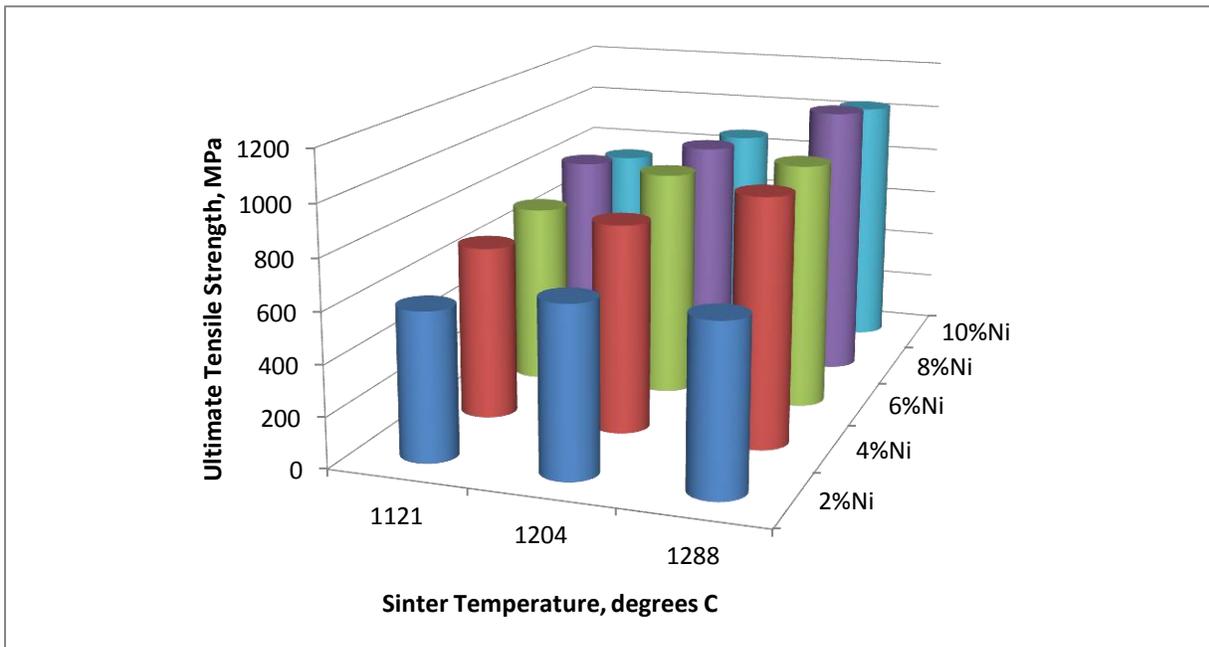
Samples were tested generally in the as-sintered condition, using industry test methods, while some were subjected to a temper/draw at 204 °C (400 °F). These samples were also tested and the data tabulated.

Mechanical Properties

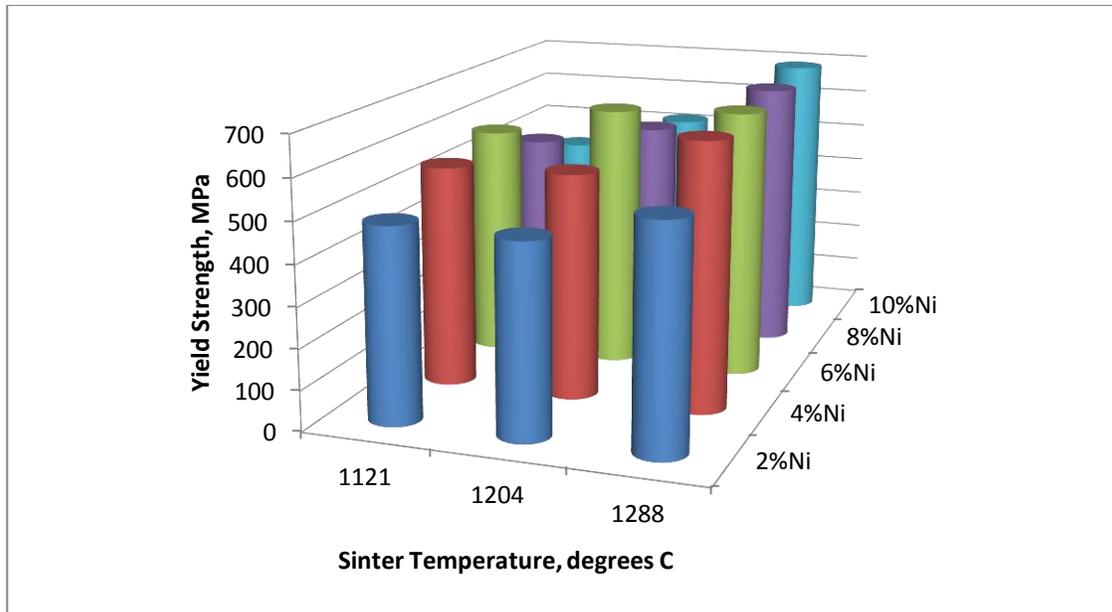
Table I below shows the averaged mechanical property data from the tensile tests, including hardness and impact tests for samples that were molded at 552MPa (40 tsi). The effect of Ni content (C content fixed at 0.65%) and sintering temperature on ultimate tensile strength (UTS) is shown in Figure 3. A similar representation of their effect on yield strength is shown in Figure 4. In like manner, their effect on impact strength is shown in Figure 5.

**Table I:** Mechanical Properties of Samples Compacted at 552 MPa (40 tsi) and Processed at Three Sintering Temperatures.

Property	2%Ni			4%Ni			6%Ni			8%Ni			10%Ni		
	1121 °C	1204 °C	1288 °C	1121 °C	1204 °C	1288 °C	1121 °C	1204 °C	1288 °C	1121 °C	1204 °C	1288 °C	1121 °C	1204 °C	1288 °C
UTS, Mpa	586	669	662	690	827	979	724	910	986	814	917	1103	738	869	1034
Yield, Mpa	483	476	552	552	558	662	579	655	669	496	552	676	427	517	690
Elongation, %	1.8	2.4	2.5	1.7	1.8	2.2	1.7	2	1.5	3	2.2	2	3	2.5	2
Hardness, HRC	2	7	HRB91	16	27	26	24	30	38	17	29	34	22	30	40
Impact, J	12	16	20	18	23	24	19	23	31	22	27	33	20	30	35
Density, g/cc	7.00	7.05	7.11	7.05	7.08	7.12	7.09	7.14	7.19	7.14	7.20	7.23	7.16	7.22	7.30

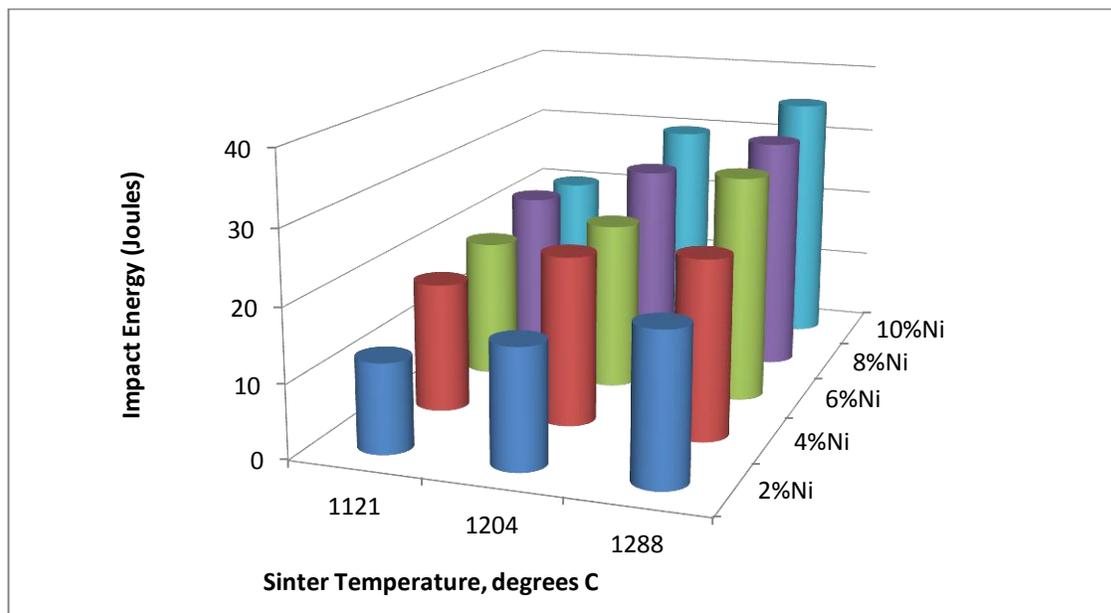


**Figure 3.** Effect of Ni content and sintering temperature on Ultimate Tensile Strength (UTS). Constant C level of 0.65%.



**Figure 4.** Effect of Ni content and sintering temperature on Yield Strength. Constant C level of 0.65%.

As one might expect, increasing sintering temperature, leads to improvements in mechanical properties (UTS, yield strength, hardness and impact strength) at any of the selected Ni levels, because of higher diffusion rates.<sup>8,16</sup> There is a general trend for higher strength with increasing Ni content and sintering temperature, which is especially evident on inspection of the graph on impact strength (Fig. 5). This is in general agreement with previous work on sinter hardened PM nickel steels.<sup>16-18</sup>

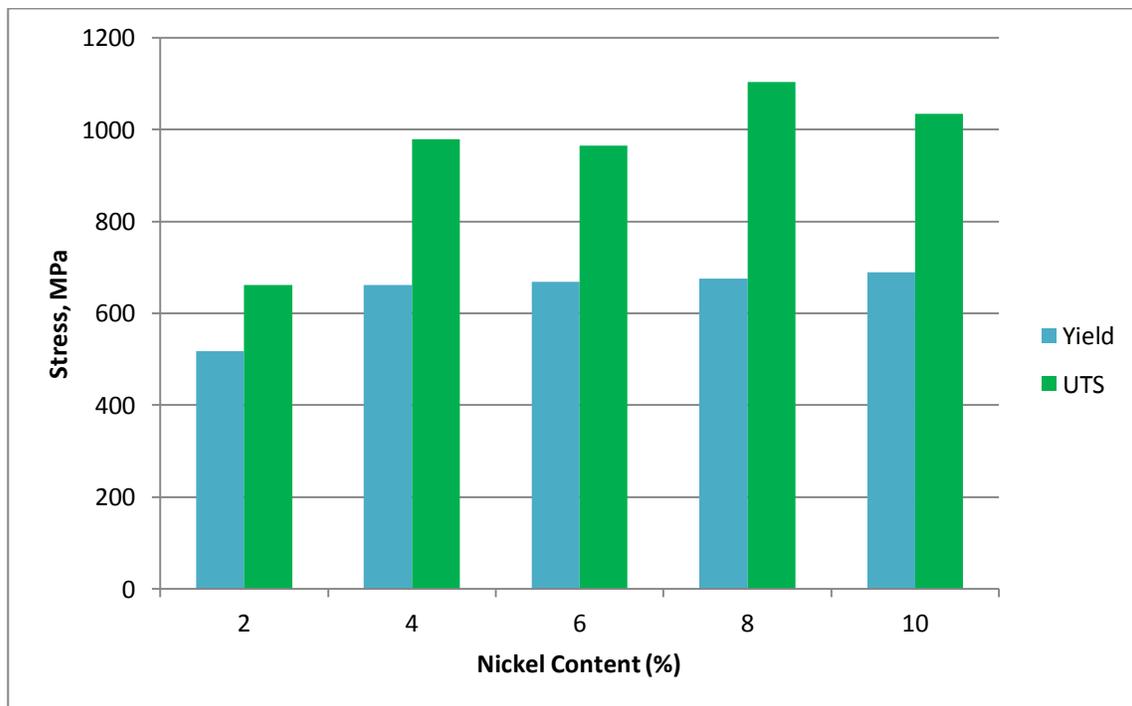


**Figure 5.** Effect of Ni Content and Sintering temperature on Impact Energy. Constant C level of 0.65%.

The high temperature study (1316 °C, 2400 °F, 75%Ni/25%N) by Hamill et al.<sup>17</sup>, looked at 4 and 6%Ni and demonstrated the excellent combination of mechanical properties that can be obtained by processing these materials at high temperatures. Causton and Fulmer<sup>18</sup> used lower sintering temperatures (1120 °C, 2050 °F, in endogas) and lower Ni levels (2%, 4%) and, consequently, observed significantly lower impact energies and lower ductility levels. Both studies also investigated the effect of adding 1 %Cu and increasing pre-alloyed Mo up to 1.50 %. There appeared to be no value of adding the extra Mo in the latter study, either in the accelerated or normal furnace cooling rates. At the 2 %Ni level the effect of 1% Cu addition was minimal at best, and even negative to UTS especially with 4 %Ni and 1.50 %Mo.

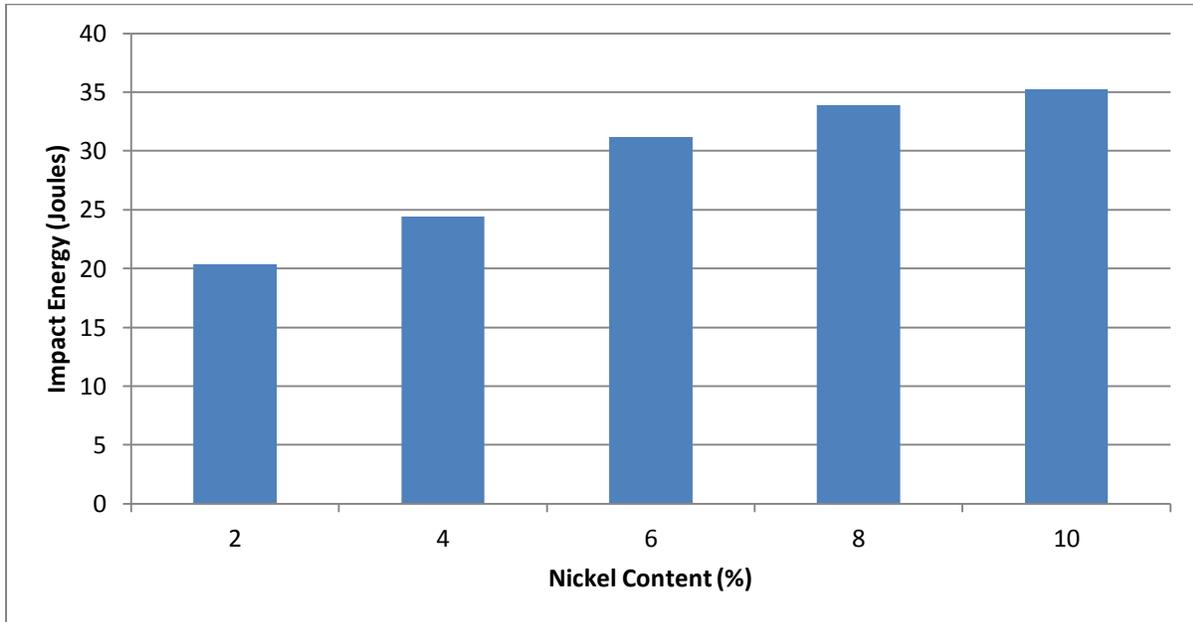
There appeared to be slightly contradictory results reported in the high temperature sinter work of Hamill et al.<sup>17</sup> With the pre-alloyed 0.5 %Mo base, the 1% Cu increased UTS for both the 4 %Ni and 6 %Ni alloys. Similarly, for the 1.5 %Mo base and 4 %Ni alloy. However, in the 1.5 %Mo base and 6 %Ni alloy, the added copper resulted in a slight drop in UTS and yield strength values. This drop in strength was attributed to the formation of voids at prior copper sites, leading to large pore formation after the high temperature sinter. A final note to this work is that they found no value to adding a tempering operation to the process in order to improve mechanical properties. They attributed this to the possibility that Ni-rich areas had sufficient ductility and that they were able to relieve the stresses that might otherwise be created in the normal course of martensite formation, during furnace cooling.

Figure 6 shows UTS and yield strength as a function of Ni content for test samples sintered at 1288 °C, (2350 °F), indicating a strong relationship of these two properties with Ni level. There is a general trend for increased yield strength with Ni level, with a plateau near 700 MPa (100 ksi, for these test samples molded at 40 tsi).



**Figure 6.** Effect of Ni content on UTS and Yield Strength; C level fixed at 0.65% and a sintering temperature of 1288 °C, (2350 °F).

For a constant 0.65% C level and a single sintering temperature of 1288 °C, (2350 °F), the impact energy has a very strong relationship to Ni content as shown in Figure 7. This can be explained by the synergistic effect of Mo and Ni in enhancing strength and, more importantly, to the increasing levels of Ni-rich areas which promote retained austenite in the microstructure.<sup>17, 19</sup> These softer areas of the heterogeneous microstructure are able to plastically deform without cracking, and absorb the externally applied impact.



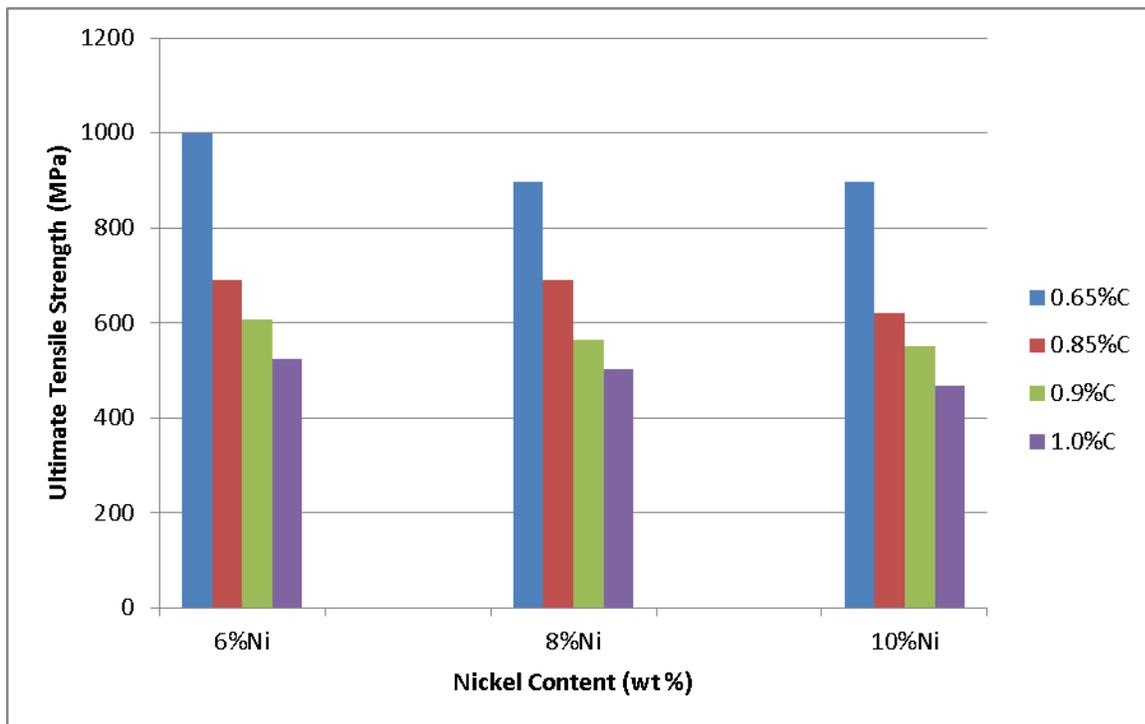
**Figure 7.** Effect of Ni content on Impact Properties; C level fixed at 0.65% and sintering temperature of 1288 °C (2350 °F),

An excellent study by Tougas et al<sup>19</sup> made the point that the retained austenite (Ni-rich areas) are expected to be at the surface of Fe powders and at the necked regions where the particles join together. Since these are softer than the martensitic regions these weaker areas should lead to weaker, not stronger, parts. They hypothesized, and proved, that stress-induced martensite was the mechanism by which nickel steels can have both increasing strength and energy absorption capability. They conducted detailed core and surface microstructural analyses and showed that the surface of the test samples exhibited more martensite than the core. Furthermore, they observed even more martensite at the ruptured edge of the test sample, where the additional stress/strain would conceptually create higher amounts of this phase.

This concept of stress/strain induced martensite formation in the Ni-rich areas is quite novel for PM nickel steels, yet is well known in other, non-ferrous, material systems. For example, the shape memory alloy NiTi<sup>20</sup> and nanocrystalline NiAl<sup>21</sup> materials, exhibit reversible stress-induced martensitic transformations. In the latter example, the phase transformation explains the 40% ductility, in a material that would otherwise be very brittle. The fact that intermetallic NiAl has an ordered B2 structure means that it does not have the required slip systems to exhibit ductility in the bulk form. In the nanocrystalline form, the material undergoes a stress induced transformation to BCT (body centered tetragonal) structure. Similar reversible stress-induced transformations were observed in the Zr-Co-Ni system (also ordered B2) and helped to explain the high ductility (70% deformation) in room temperature tests.<sup>22</sup>

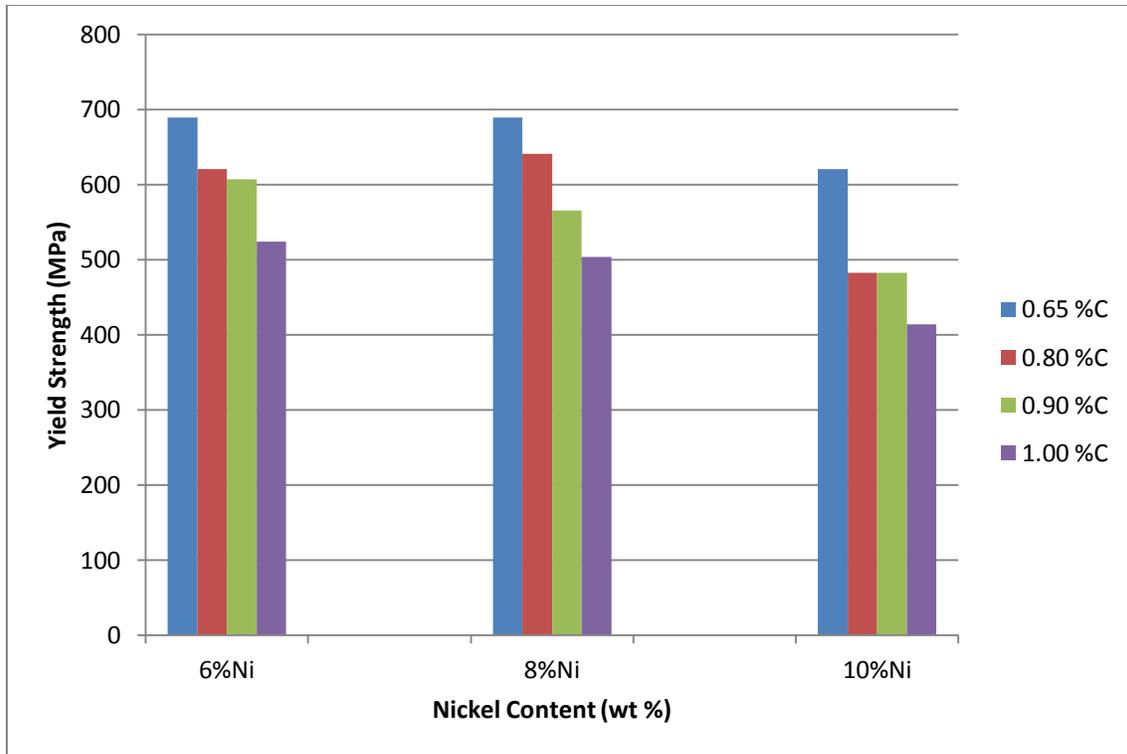
Using a similar base Fe-0.85%Mo alloy to the current study, Marucci and Rawlings<sup>23</sup> presented mechanical property data for FL-4405 (pre-alloyed 0.85%Mo, 0.6%C, No Ni) and FLN2-4405 (same, with 2%Ni added) at various levels of density, including in the full-dense, forged, condition. UTS and Yield values in the sinter hardened condition for FL-4405 were about 862 MPa (125 ksi) and 586 MPa (85 ksi), while the corresponding values for the FLN2-4405 were about 1200 MPa (174 ksi) and 772 MPa (112 ksi), respectively.

Figures 8 and 9 show the effect of increasing Ni and C levels on the ultimate tensile strength and yield strength, respectively, on these nickel steels. The decreasing strength values indicate that the eutectoid composition has been exceeded for the higher Ni levels. This is in general agreement with the work of Gelinas and St. Laurent<sup>24</sup> for Fe-4%Ni-1%Cu (base of pre-alloyed 0.85%Mo) formulated to various C levels from 0.3 to 0.9%C and sintered at 1121 °C (2050 °F). Their data for samples in the as-sintered condition showed that the highest UTS value was achieved at a C level between 0.5 and 0.55%.

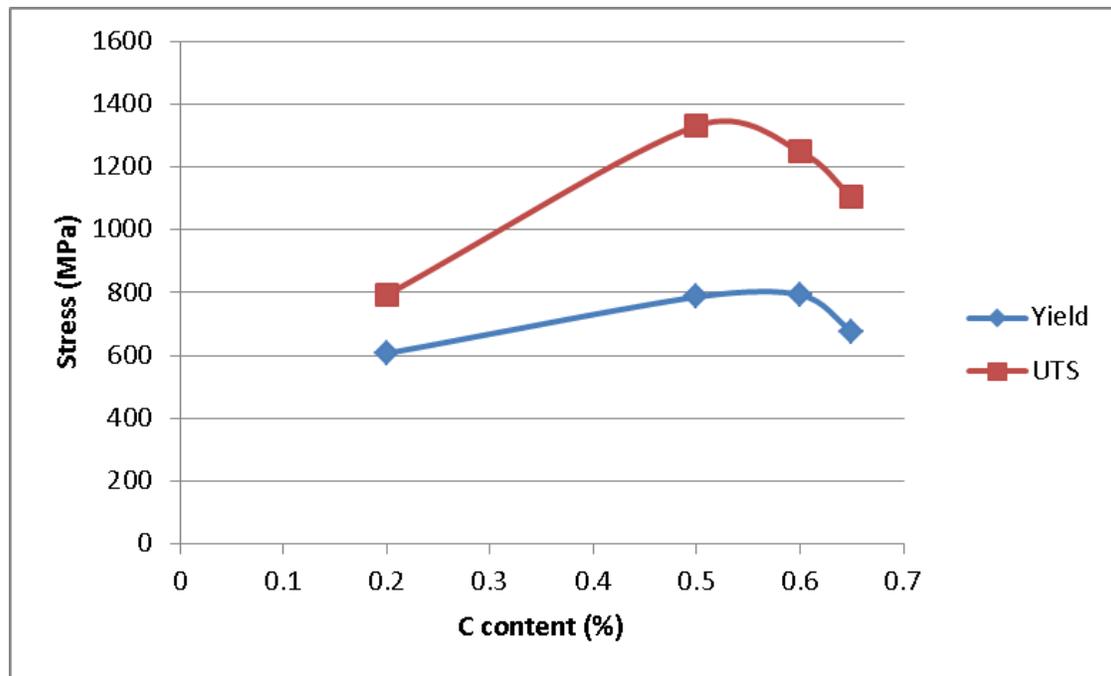


**Figure 8.** Effect of Ni and C level on UTS after sintering at 1274 °C (2325 °F).

When compared to the work of Hamill et al<sup>17</sup>, the current study reflects lower strength values. We believe this difference is due to processing differences as well as the fact that the C level is too high. In order to check this concept out, additional test samples were molded at 50 tsi using an 8%Ni alloy and varying the C level from 0.2 % to 0.6 5%C. Sintering was performed in pusher furnaces at a temperature of 1288 °C (2350 °F). The tensile test results are plotted in Fig 10.



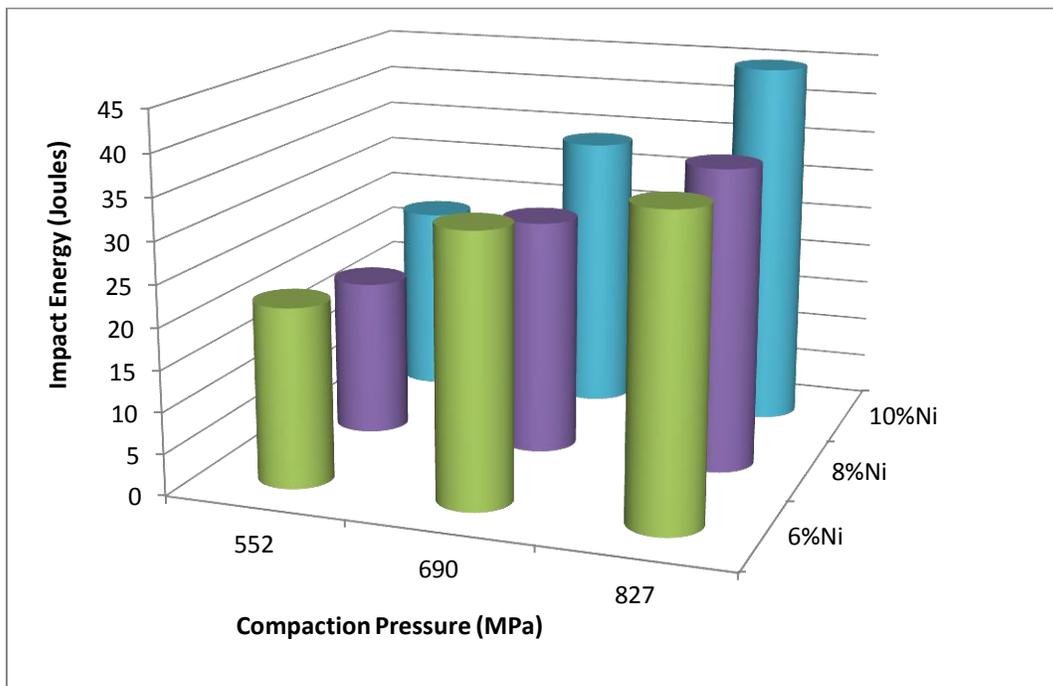
**Figure 9.** Effect of Ni and C level on yield strength after sintering at 1274 °C (2325 °F).



**Figure 10.** Effect of C level on the UTS and yield strength; Ni level fixed at 8%. The 0.65% C test samples were molded at 552 MPa (40 tsi), while all others were molded at 690 MPa (50 tsi).

The optimum strength is clearly attained with about 0.5%C. UTS values near 1380 MPa (200ksi) combined with an elongation of over 2% and an impact energy of over 34 Joules (25 ft. lbs.) is achievable with a single press and single sinter processing methodology. Furthermore, with an apparent hardness of about 30 HRC (particle hardness above 55 HRC) at a density near 7.2 g/cc, the resulting product is quite suitable for high strength applications requiring significant resistance to wear. These results are quite consistent with the values attained by Hamill et al.<sup>17</sup>

With this knowledge that 0.5 %C is close to the optimum level for the higher Ni containing alloys being studied here, one additional dependency was studied and documented. The effect of compacting at 552 MPa (40 tsi), 689 MPa (50 tsi), and 827 MPa (60 tsi) on impact energy is shown in Figure 11. The sintering temperature was kept constant at 1288 °C (2350 °F), as was the C level at 0.5 %. As might be expected, the impact energies increase quite nicely with increasing compaction pressure. Furthermore, increasing Ni content leads to improving energy absorption; with 10 %Ni and a compaction pressure of 827 MPa (60 tsi), the impact energy is an impressive 45 Joules (33 ft. lbs.). Recall that this is in combination with an equally impressive tensile strength of nearly 1380 MPa (200 ksi), as shown earlier in Figure 10.



**Figure 11.** Effect of Compaction Pressure and Ni content on Impact Energy. Constant sintering temperature (1288 °C, 2350 °F) and C content (0.5 %).

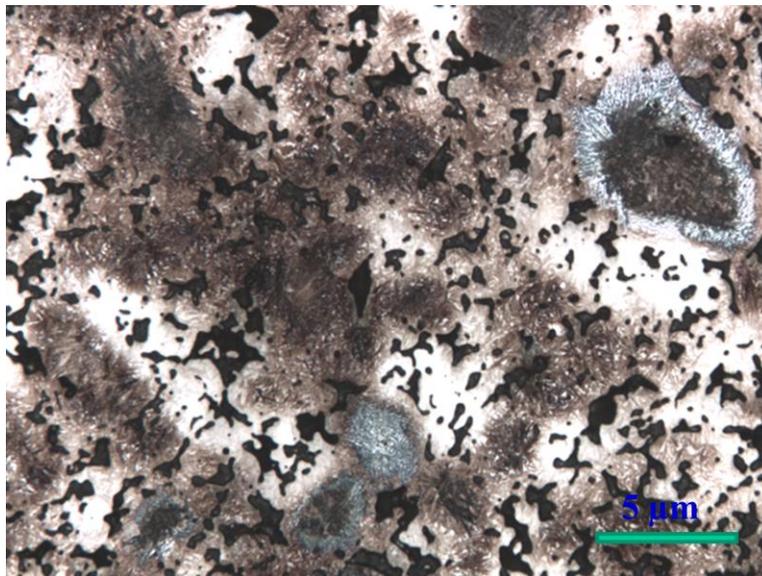
Consideration should be given to the use of Ni contents in the range of 6-10%, in combination with high temperature sintering and optimization of %C, to achieve a combination of high strength and ductility/toughness. The higher levels of Ni must be allowed to diffuse into the base Fe-0.85 %Mo alloy powder (hence the need for high temperature sintering), but at the same time, some excess Ni is necessary to form the Ni-rich austenite phase which is subsequently able to transform to martensite during deformation. Note that products processed in this way result in more than double the impact and ductility values of lower Ni alloys. Addition of admixed Cu is to be avoided as this may lead to voids, which decrease these desirable mechanical properties.

## Metallography

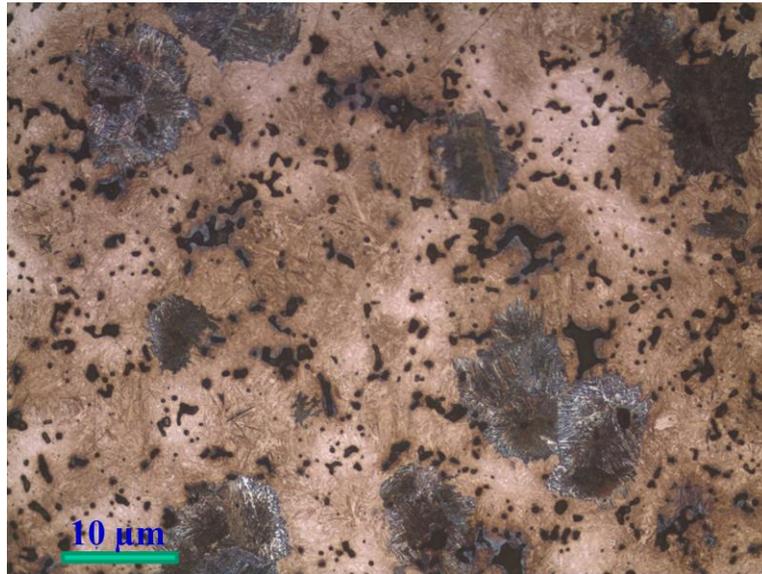
Metallographic examination of the sintered test samples reveals the microstructural phases and features that are responsible for the mechanical properties observed for these alloys. Figure 12, shows a typical microstructure of the Fe-8 %Ni-0.5 %C-0.85 %Mo alloy, with primarily martensite, bainite and pearlite. The lightly etched areas are so-called nickel rich areas (NRA) and, perhaps, untransformed austenite. We make this qualified statement, because further etching reveals that the light areas also contain martensite, as well as some austenite in the background. We interpret the lamellar phase to be pearlite, as against bainite. This heterogeneous microstructure is the key to having good strength along with good toughness. The martensitic areas have an apparent hardness near HRC 30, while the particle or micro-hardness is in the HRC 55-60 range in the martensitic areas. After detailed examination of the metallographic samples of this composition, the conclusion was that no carbides were visible.

Figure 13 is similar in composition to that of Figure 12, except that the C level has been increased from 0.5% to 0.65%, and the sample has been etched a little darker. Note that there are lighter nickel rich areas here also, but martensite is visible in these areas. No cementite was found in this sample either.

As the carbon level was increased to 0.8%C, some carbides were seen, but on very rare occasions (Figure 14). When 0.9%C was used, carbide networks were readily visible, as shown by the micrographs in Figure 15. When 10%Ni is used, the carbide networks are readily visible even at 0.8%C, but are harder to locate when the carbon content is at 0.65% (Figure 16). These carbides, of course, lead to embrittlement as exhibited by the decrease in tensile elongation, as well as premature failure.



**Figure 12**, Typical microstructure of the Fe-8%Ni-0.5%C-0.85%Mo alloy; consisting of martensite, lightly-etched nickel-rich areas, upper/lower bainite and pearlite. Original magnification 200x. 2 % nital etch.

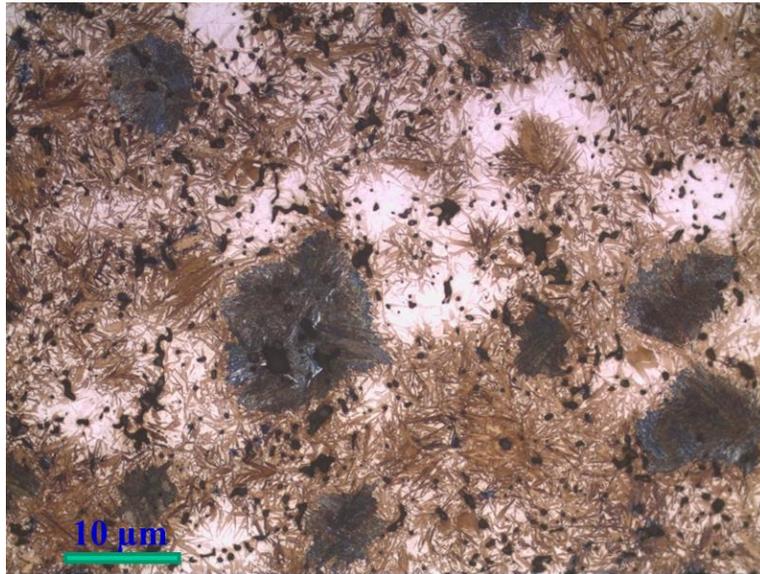


(a) Original mag. 200x

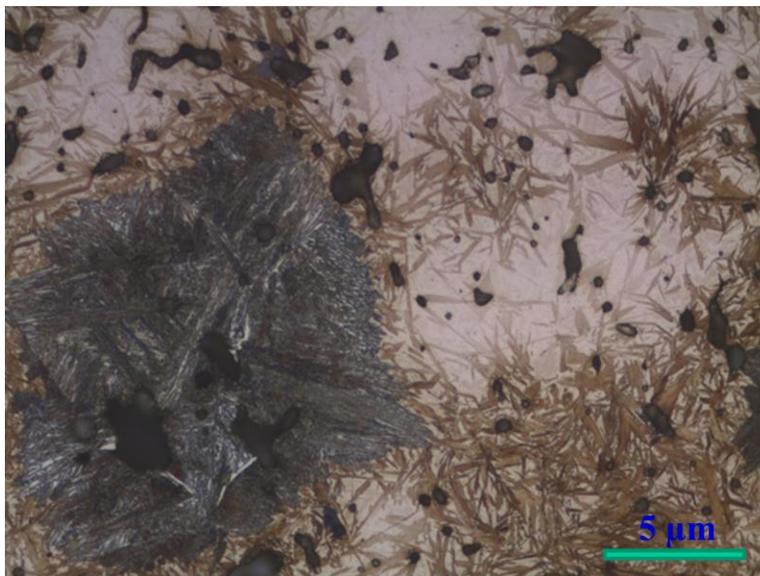


(b) Original mag. 500x

**Figure 13**, Typical microstructure of the Fe-8%Ni-0.65%C-0.85%Mo alloy; consisting of martensite, lightly-etched nickel-rich areas, upper/lower bainite, and pearlite. 2 %nital etch.

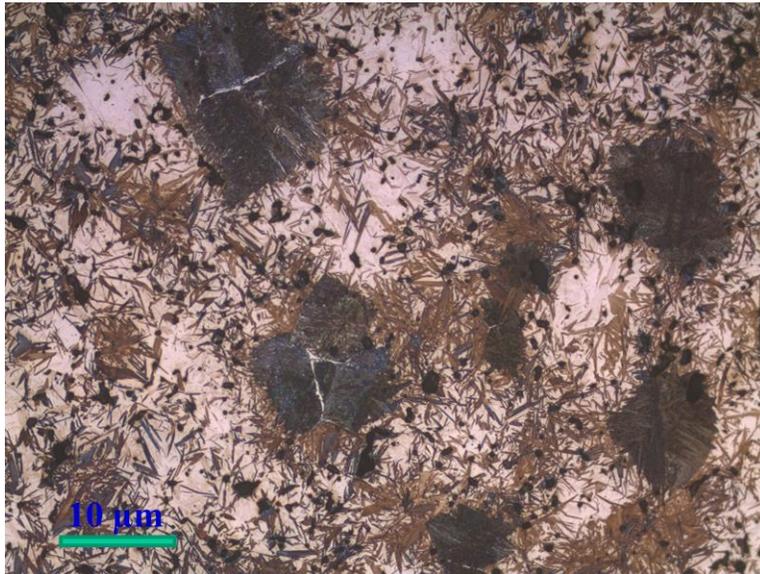


(a) Original mag. 200x

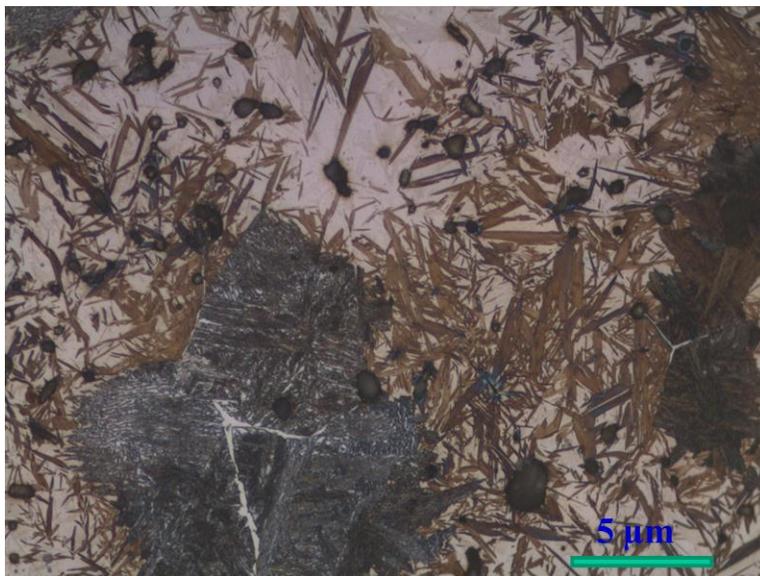


(b) Original mag. 500x

**Figure 14**, Typical microstructure of the Fe-8%Ni-0.8%C-0.85%Mo alloy; consisting of martensite, lightly-etched nickel-rich areas, upper/lower bainite and pearlite. Rare signs of carbides in the pearlitic/bainitic region. 2 % nital etch.

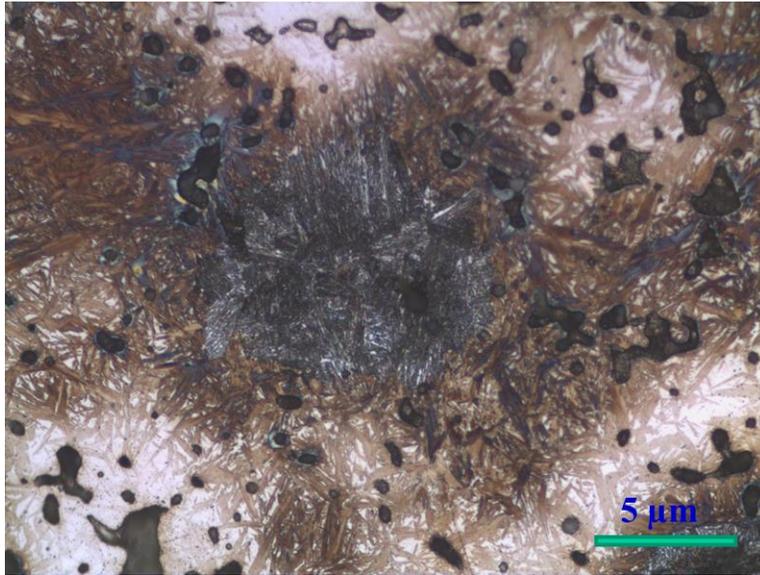


(a) Original mag. 200x

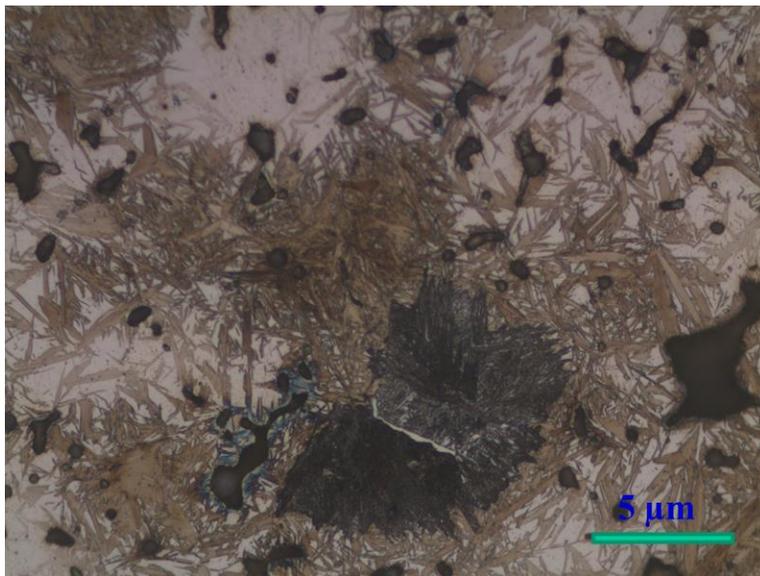


(b) Original mag. 500x

**Figure 15**, Typical microstructure of the Fe-8%Ni-0.9%C-0.85%Mo alloy; consisting of martensite, lightly-etched nickel-rich areas, upper/lower bainite and pearlite. Carbide networks clearly visible. 2 %nital etch.

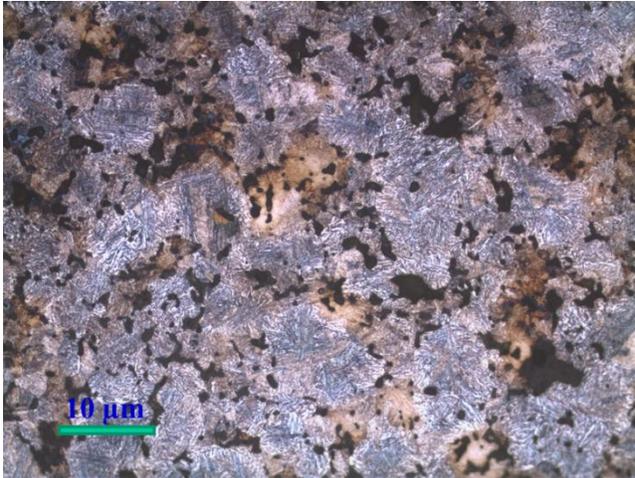


(a) 0.65 %C. Original mag. 500x

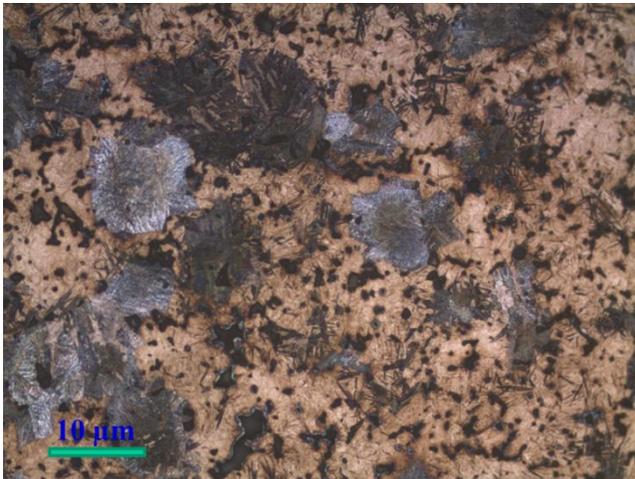


(b) 0.80 %C. Original mag. 500x

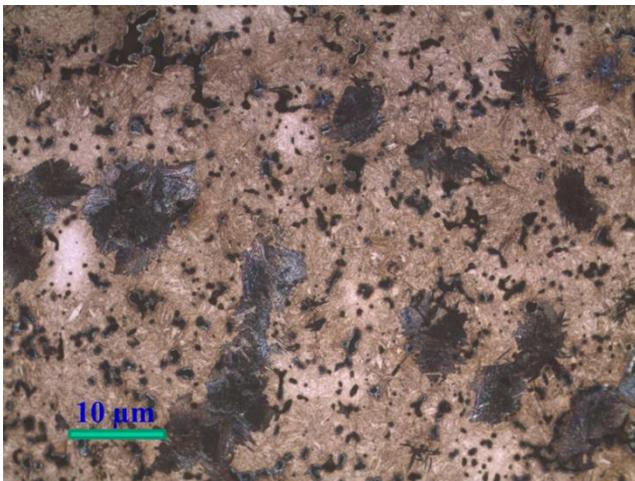
**Figure 16**, Typical microstructure of the Fe-10%Ni-0.85%Mo base alloy; consisting of martensite, lightly etched nickel-rich areas, upper/lower bainite and pearlite. Carbide difficult to find in the 0.65 %C alloy (a), but readily visible in the 0.80 %C alloy (b). 2 % nital etch.



(a) 2 %Ni Original mag. 200x



(b) 4 %Ni. Original mag. 200x



(c) 6 %Ni. Original mag. 200x

**Figure 16**, Typical microstructure of the Fe- XX %Ni- 0.65%C-0.85%Mo base alloy; consisting of martensite, lightly-etched nickel-rich areas, upper/lower bainite and pearlite. Note the increasing level of martensite and decreasing level of pearlite as Ni-content is increased. 2 % nital etch.

As a final note on the effect of Ni, Figure 16 demonstrates how the microstructure changes; there is much less martensite but much more pearlite at the lower Ni contents. This explains why strength levels are so low at low Ni levels, when test samples are evaluated in the as-sintered condition.

## CONCLUSIONS

The primary highlights of this study are the following

1. Using a pre-alloyed base powder containing 0.85% Mo (which aids hardenability during sinter hardening), mechanical properties increase with increasing Ni content.
2. Increasing sintering temperature increases mechanical properties, and is a necessity for obtaining the best value from the Ni additive.
3. The high temperature sintering is especially valuable to maximizing impact energy and ductility.
4. The optimum C level for the 6 to 10% Ni alloy is about 0.5%.
5. The heterogeneous microstructure attained with these high nickel alloys results in a combination of mechanical properties that compete well with other PM alloys that must be processed to greater than 7.4 g/cc.
6. Using a single-press and single-sinter processing protocol, a high temperature sintering approach can provide the following combination of properties: UTS near 1380 MPa (200 ksi), yield strength over 827 MPa (120ksi), elongation over 2 %, impact energy over 40 Joules (30 ft. lbs.), and a density near 7.3 g/cc.

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