

# **MICROSTRUCTURE AND MECHANICAL PROPERTIES OF A BAINITIC PM STEEL**

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## **ABSTRACT**

The microstructures of PM steels are typically pearlitic (predominately in the as sintered condition) or martensitic (occurring as the result of accelerated cooling or heat treatment). By design of the alloy system, a predominately bainitic microstructure can be produced in a PM steel after sintering. In the wrought ferrous industry, bainitic steels are known to exhibit high strength and hardness and also a high level of toughness. The aim of this research was to develop a bainitic microstructure in a PM steel that exhibits a high level of strength and toughness with acceptable ductility. The study focuses on alloy composition and attendant mechanical properties and microstructure. In addition, processing routes to achieve bainitic and mixed (pearlitic/bainitic/martensitic) microstructures with the potential to enhance mechanical properties are reviewed.

## **INTRODUCTION**

PM parts producers continue to explore methods to achieve enhanced mechanical properties while utilizing lower cost alloys or lower amounts of alloying elements. Techniques such as micro-alloying, precipitation-hardening and secondary hardening have been used to accomplish this goal. Recently, wrought steels with a predominately bainitic microstructure that exhibit a high strength with excellent toughness and ductility have been developed. These steels are produced utilizing lower alloy contents than used in predominantly martensitic steels [1-7].

Combining alloy design with direct cooling leads to a fine bainitic microstructure or mixtures of bainite and martensite that can be used in place of quenched and tempered (Q&T) steels. Bainite has always been considered a “troublesome” microstructure in PM steels. Many times in PM steels the formation of bainite is commonly caused by local variations in alloy content and/or cooling rates. Small amounts of bainite in a pearlitic microstructure lead to unwanted hard spots while small amounts of bainite in a martensitic microstructure leads to soft spots, both conditions leading to inferior properties. In these situations the bainite is coarse and is normally called upper bainite.

With the advent of accelerated cooling in the sintering furnace the possibility of producing a PM steel with high levels of fine bainite or mixtures of fine bainite and martensite now appears possible. For a microstructure consisting of fine bainite, strength follows the Hall-Petch relationship in which the strength is inversely proportional to the grain size. For a mixed microstructure the steel has a high resistance to cleavage fracture and void formation due to the absence of coarse eutectoid carbides, thus simultaneously increasing strength and toughness. In fact, improvements to impact toughness and ductility can be obtained in martensitic steels by introducing small amounts of lower bainite.

Bainite is formed over a wide range of temperatures and the microstructure varies. There are essentially two types of bainite: upper bainite formed by cooling from austenite into the temperature range of 300 °C to 500 °C, and lower bainite which forms by cooling to the temperature range of 200 °C to 300 °C. The general definition of bainite is that the microstructure consists of a non-lamellar mixture of ferrite and carbides. More specifically, upper bainite consists of lath or plate shaped ferrite arranged in packets separated by layers of cementite particles (Figure 1). Lower bainite consists of fine needlelike plates or laths with the carbides precipitated within the laths. The reason for this differentiation is that there are clear differences between the mechanical properties of upper and lower bainite. Any refinement of microstructure in low alloy steels normally leads to an increase in the properties, particularly the strength, hence making lower bainite the more attractive microstructure.

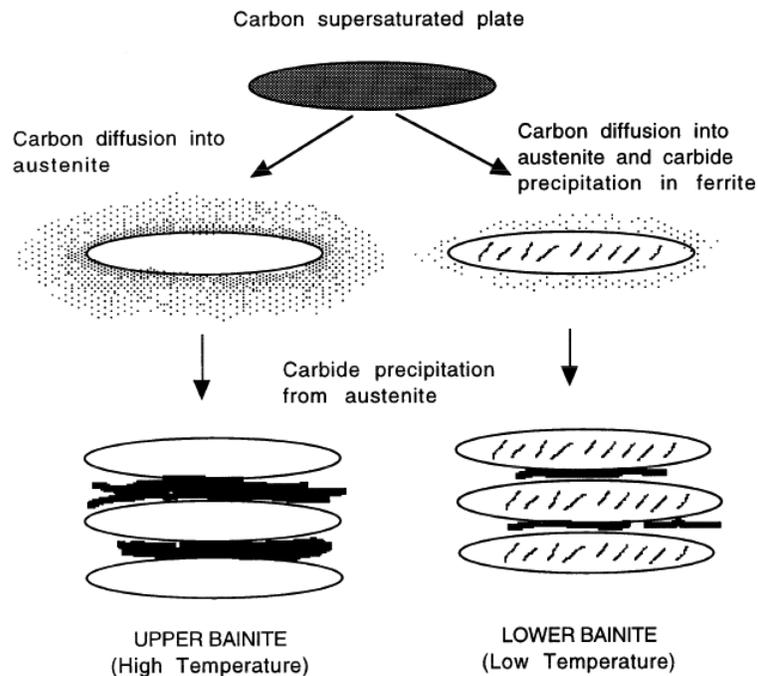


Figure 1: Schematic representation of upper and lower bainite [8].

The kinetics of bainite formation vary with alloy content. Depending on the alloy content, cooling rates in the range  $\sim 1\text{-}100\text{ }^{\circ}\text{C/s}$  can allow the formation of bainitic microstructures. There has been a recent trend in PM alloys to utilize alloying elements that provide enhanced hardenability: examples are chromium, manganese, silicon and vanadium [9-11]. The higher sintering temperatures allow for the reduction of oxides from these elements in atmospheres that are predominately nitrogen with small amounts of hydrogen (90 v/o nitrogen / 10 v/o hydrogen). The effect on these alloy elements on the formation of bainite is shown in continuous cooling transformation diagrams, Figure 2. Figure 2(a) shows a plain carbon steel ( $\sim 0.40\text{ w/o C}$ ) with only additions of manganese ( $\sim 0.70\text{ w/o}$ ). It can be seen that cooling rates in the range of  $100\text{ }^{\circ}\text{C/s}$  are necessary to form bainite in this alloy composition. This cooling rate is also not achievable with current sinter hardening furnaces. However if alloying elements of (chromium, molybdenum and vanadium) are added to the same steel (addition levels of 1.19, 0.50 and 0.22 w/o respectively), the cooling rate necessary to achieve bainite during continuous cooling is reduced. In this alloy, cooling rates typically found in sintering furnaces with accelerated cooling ( $0.7\text{ to }3\text{ }^{\circ}\text{C/s}$ ), are sufficient to form bainite. If the cooling rate is sufficiently high, predominately lower bainite will form.

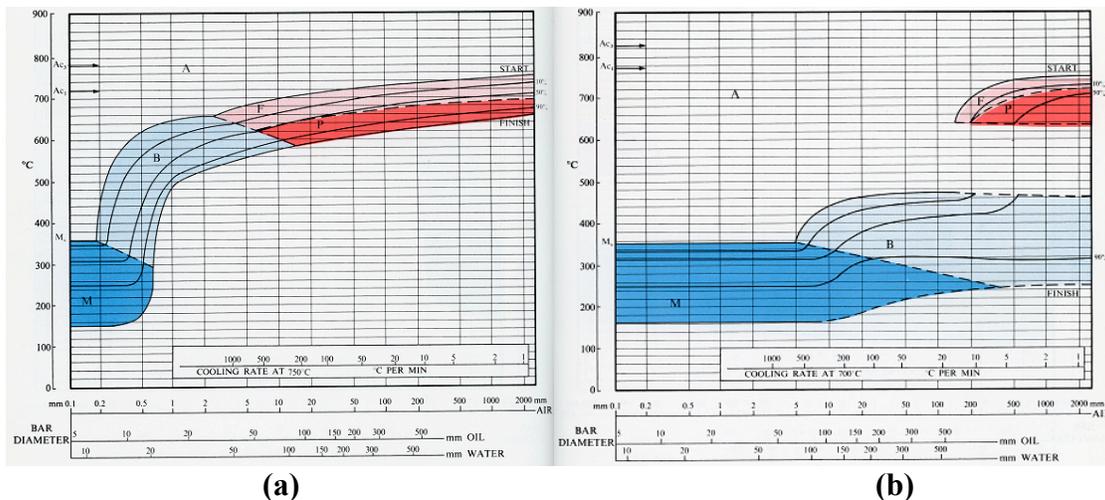


Figure 2: CCT curves for (a) a plain carbon steel and (b) steel with similar composition to (a) but with the additions of chromium, molybdenum and vanadium [12].

Figure 3 shows the properties of some novel bainitic steels in comparison with some high performing steels such as martensitic, high strength low alloy, dual phase and conventional mild steels. It can be seen that this new class of bainitic steels exhibit exceedingly high strength levels while retaining ductility exceeding those of martensitic steels. In general, bainitic steels are ideal for applications requiring strengths under 1000 MPa, where the total alloy concentration is kept below 2 w/o [8]. However, the design of the alloy must be considered along with the thermal treatment to obtain the correct microstructures. PM steels with adequate hardenability must be designed, especially considering the lower cooling rates currently available in PM compared with wrought alloys.

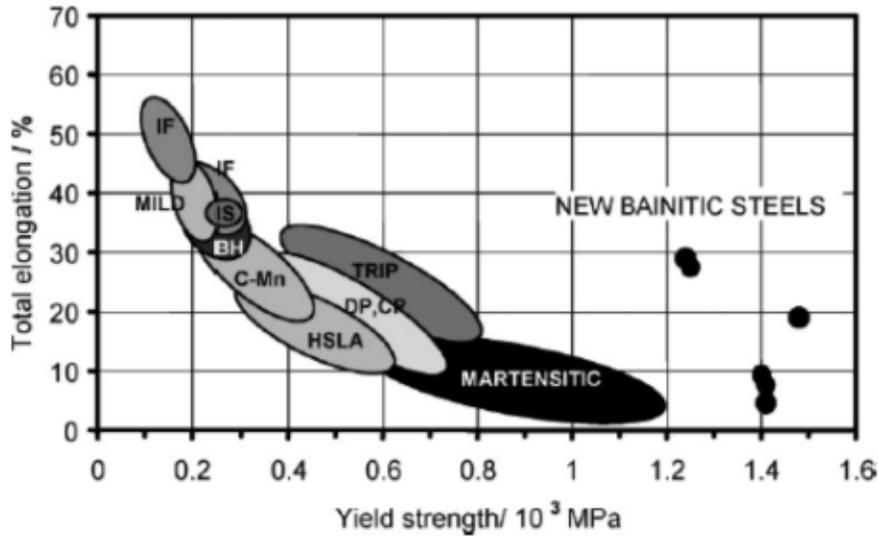


Figure 3: Comparison of yield strength and elongation for high performing versus new class of bainitic steels (wrought alloys) [2].

The objective of the present study was to develop an alloy that, utilizing accelerated cooling in the sintering process, would contain large amounts of lower bainite. And further to evaluate the mechanical properties of this mixed microstructure in relation to upper bainite and fully martensitic microstructures.

## ALLOY PREPARATION AND TESTING

Mixtures of the base powder and a master alloy containing silicon were utilized to prepare test specimens. The base powder was an alloy pre-alloyed with chromium, manganese, molybdenum and vanadium. The master alloy was 75 w/o ferro-silicon with a mean particle size ( $d_{50}$ ) of approximately 10  $\mu\text{m}$ .

The powders were mixed with Acrawax C lubricant and graphite. Graphite additions were 0.60 w/o (unless otherwise noted) resulting in a sintered carbon level of approximately 0.50 w/o. Samples for transverse rupture (TR), impact testing and tensile testing were compacted uniaxially at a pressure of 690 MPa. The test pieces were sintered in a high temperature Abbott continuous-belt furnace at 1260 °C (2300 °F) for 30 min in an atmosphere of 90 v/o nitrogen / 10 v/o hydrogen. Accelerated cooling experiments were conducted using the same furnace but utilizing a gas quench to provide accelerated cooling at rates ranging from 0.7 to 2.4 °C/s. Test pieces were tempered at temperatures from 204 °C (400 °F) to 760 °C (1400 °F) for 1 h in 100 v/o nitrogen.

For heat treatment, samples were austenitized at 900 °C (1650 °F) for 60 min at temperature in a 75 v/o nitrogen / 25 v/o hydrogen atmosphere prior to quenching in oil.

Prior to mechanical testing, green and sintered densities, dimensional change (DC), and apparent hardness were determined on the tensile and TR samples. Five tensile specimens and five TR specimens were evaluated for each composition. The densities of the green and sintered steels were determined in accordance with MPIF Standard 42. Tensile testing followed MPIF Standard 10 and apparent hardness measurements were made on the tensile and TR specimens, in accordance with MPIF Standard 43.

Green density of the base powder was measured with a 12.7 mm diameter die at a height of 12.7 mm at a compaction pressure of 400 MPa with 1% ZnSt.

The apparent density and flow rate of the base powder were determined in accordance with MPIF Standards 3 and 4, and the sieve analysis in accordance with MPIF Standard 5.

Specimens for microstructural characterization were prepared using standard metallographic procedures. Subsequently, they were examined using light optical and scanning electron microscopy in the polished and etched conditions. Light optical microscopy was used to examine the microstructures in the etched (2 v/o nital / 4 w/o picral) condition using multiple magnifications. Quantitative analysis using a manual point count on etched specimens (1 v/o nital / 4 w/o picral) was performed at 1000x to estimate the proportions of transformation products. Multiple fields were sampled on each prepared cross-section using an x-y grid containing 165 equally spaced points. Additionally, microindentation hardness measurements were performed on lightly etched (2 v/o nital / 4 w/o picral) specimens.

The scanning electron microscope was used to image both the etched microstructures and fracture surfaces from impact tested specimens.

## **RESULTS AND DISCUSSION**

### **Alloy System**

The chemical composition of the alloy system was chosen primarily to minimize cost and to have enough alloying content to ensure that a bainitic microstructure could be formed utilizing a conventional sinter-hardening furnace. Based on previous work by the authors [13] it was determined that elements such as chromium, manganese and silicon can have a synergy with molybdenum and lead to a hardenable alloy at a reasonable cost. Molybdenum is known to enhance bainite formation while retarding the formation of pearlite/ferrite so it was chosen as the base element from which to build the alloy. The addition of vanadium also increases the hardenability of low alloy steels and has the advantage that it combines with nitrogen from the sintering atmosphere to form vanadium nitride precipitates which limit the austenite grain size during sintering leading to a finer microstructure upon cooling. It has also been found that both silicon and vanadium suppress the formation of carbides which favors the formation of bainite. Chromium, molybdenum, silicon and vanadium are strong carbide forming elements and undergo secondary hardening during tempering at high temperatures. This hardening occurs when

fine alloy carbides form at the expense of the cementite and generally occurs at temperature ranges from 500 to 600 °C. This secondary hardening (or age-hardening) often results in strength levels in excess of what could be achieved in as-quenched martensite.

Table I: Chemical Composition and Physical Properties of Experimental Alloy.

<b>C</b>	<b>S</b>	<b>O</b>	<b>N</b>	<b>Si</b>	<b>Cr</b>	<b>Mn</b>	<b>Mo</b>	<b>V</b>
(w/o)	(w/o)	(w/o)	(w/o)	(w/o)	(w/o)	(w/o)	(w/o)	(w/o)
0.010	0.010	0.250	0.020	0.75	0.58	0.40	0.89	0.10

<b>+80</b>	<b>+100</b>	<b>+140</b>	<b>+200</b>	<b>+230</b>	<b>+325</b>	<b>Pan</b>	<b>AD</b>	<b>Flow</b>	<b>GD</b>
(w/o)	(w/o)	(w/o)	(w/o)	(w/o)	(w/o)	(w/o)	(g/cm <sup>3</sup> )	(secs)	(g/cm <sup>3</sup> )
0.2	3.5	14.8	21.6	12.0	18.8	29.1	2.93	27.3	6.54

The powder was produced by conventional water atomization followed by annealing in 100 v/o hydrogen at 1000 °C. The sieve analysis, apparent density and green density of the base powder (Table I) are typical of base powders commonly used in the PM industry. Silicon was added to the powder via a master alloy and graphite was utilized for the carbon addition. All other elements were pre-alloyed into the powder prior to atomization. Carbon additions were limited to ranges of 0.40 to 0.60 w/o in preliminary studies to achieve a significant amount of bainite in the microstructure. Above this range, the carbon can lead to coarse cementite particles that act as initiation sites for fracture. For the data presented here, graphite additions of 0.60 w/o were determined to be optimum.

### Sintering Response

Initial sintering studies were needed to generate a series of microstructures containing different levels of bainite for testing. An Abbott high temperature belt furnace was utilized and a sintering temperature of 1260 °C (2300 °F) was utilized in an attempt to get as much of the silicon master alloy in solution as possible. A mixture of 90 v/o nitrogen and 10 v/o hydrogen was used for the sintering atmosphere. The furnace was equipped with a “Varicool Unit” that allowed nitrogen gas to cool the test specimens as they entered the cooling zone of the furnace. The variable speed blower allows different rates of convective cooling to be achieved. Previous measurements on this furnace showed cooling rates from 0.7 °C/s to 2.4 °C/s.

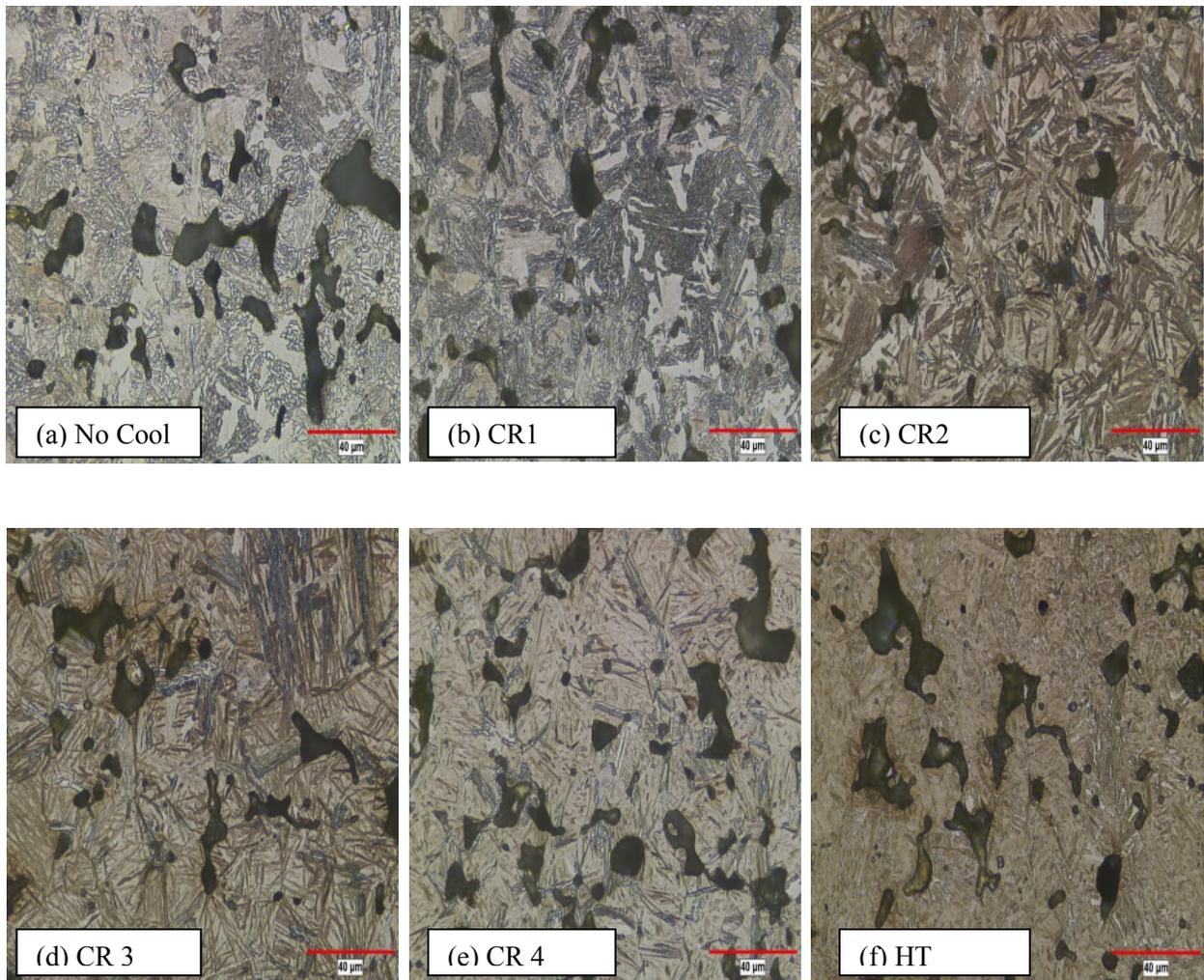


Figure 3: Microstructures of experimental alloy with various cooling rates ((a)-(e)) and austenitized and oil quenched (f).

Figure 3 shows the microstructure from the tensile specimens sintered at 1260 °C (2300 °F) and cooled at various rates. Figure 3(a) is the microstructure with only furnace cooling; i.e. no accelerated cooling utilized. The microstructure is predominantly upper bainite with plates of ferrite about 10 μm long and less than 1 μm thick. As when the accelerated cooling is utilized at a very low rate the microstructure is still upper bainite but it is finer and is difficult to resolve the individual subunits. There is a small amount of martensite in highly allowed areas, most likely due to insufficient diffusion time for full homogenization. Increasing the accelerated cooling further (CR2), the microstructure becomes predominately bainite (77%) with significantly less ferrite. However at this cooling rate, significant amounts of martensite are formed resulting in a mixed microstructure. As the cooling rate is further increased (CR3 to CR4), the amount of lower bainite decreases and the amount of martensite increased and the ferrite vanished. Figure 3(f) shows the same alloy austenitized at 900 °C and oil quenched. This microstructure consists of 100 v/o martensite and will be used as a benchmark for

comparison of the mechanical properties of the bainitic-containing alloys. A summary of the microstructures of the six samples, as shown in Figure 3, is detailed in Table II.

Table II: Summary of Microstructural Constituents of Specimens (a) to (f) in Figure 3 (values are volume percent).

Process Condition	Lower Bainite	Martensite	Upper Bainite
As Sintered (No Cool)	0	Some small amounts in Alloyed Regions	~100
Cooling Rate 1 (CR1)	0	Some small amounts in Alloyed Regions	~100
Cooling Rate 2 (CR2)	77	22	1
Cooling Rate 3 (CR3)	41	58	1
Cooling Rate 4 (CR4)	3	96	1
Heat Treated (HT)	0	100	0

## Mechanical Properties

Although a microstructure consisting of 100% lower bainite was not achieved utilizing the processing and alloying parameters used in this study, it has been found that a mixed microstructure (bainite and martensite) can provide an ideal combination of properties. Due to the absence of coarse eutectoid carbides, the steel has a high resistance to cleavage fracture and void formation. This leads to a concurrent increase in strength and toughness. Tensile test results of the alloys cited in Table II are shown in Figure 4. Figure 4 show the properties of the same alloy ranging from one of upper bainite with extensive ferrite to first one of mainly lower bainite and finally to fully martensitic (austenitizing and oil quenching). The as sintered material which consists of nearly 100 percent upper bainite has the lowest ultimate tensile strength and hardness. Upper bainite forms at relatively high temperatures (just below the range for pearlite formation) with lower hardness values than lower bainite or martensite. Upper bainite forms in blocky patches containing plates of ferrite with relatively coarse particles of cementite between the ferrite blocks. The microindentation hardness of the mixed upper bainite and ferrite (Figure 3(a)) was measured to be 335 HV<sub>100gf</sub> as compared with 750 HV<sub>50gf</sub> for the all martensitic structure. This relatively soft and coarse microstructure leads to lower strength and hardness. The same situation exists in the sample utilizing cooling rate CR1. Although the microstructure of this material is still upper bainite, the microstructure is visibly finer than in the material with accelerated cooling (Figure 3(a) versus Figure 3(b)) and this leads to an increase in strength and hardness. As the cooling rate is increased further (CR2 and CR3), lower bainite starts to form along with a significant percentage of martensite. At the highest cooling rate utilized in the study (CR4), the microstructure

becomes predominately martensitic. As a reference point, a heat treated sample was included in the study to provide a fully

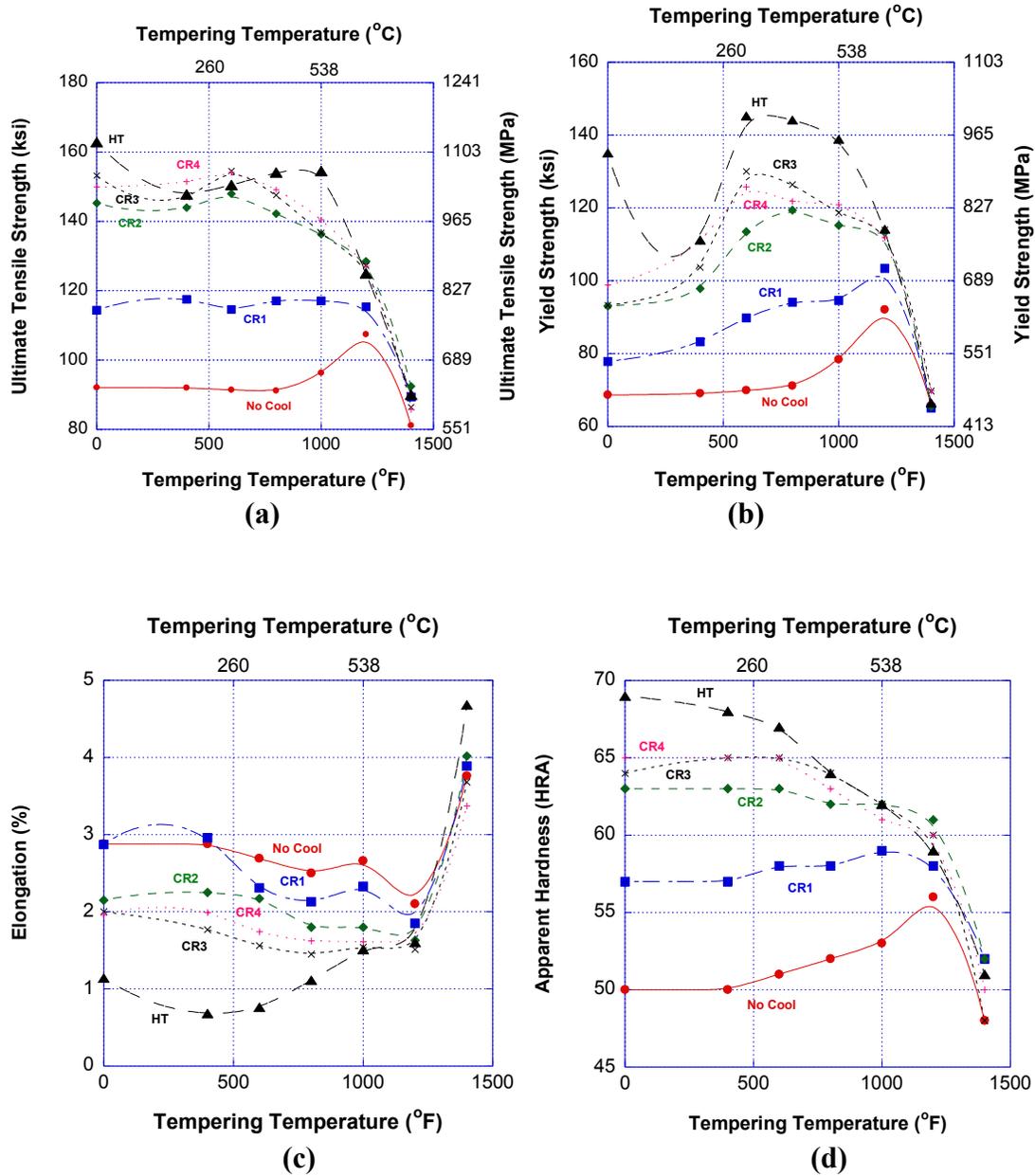


Figure 4: Tensile properties and apparent hardness of alloys shown in Figure 3. Final sintered density  $6.95 \text{ g/cm}^3$ .

martensitic structure for comparison of strength and hardness. The samples which contain a significant amount of lower bainite have ultimate tensile strengths significantly higher than those of the same alloy with upper bainite (~ 1000 MPa versus 655 MPa). Lower

bainite forms at much lower temperatures than upper bainite and develops a plate-like morphology of ferrite. Also the carbides are extremely fine particles located within the plates and are not usually discernable by optical microscopy. Figure 5 shows scanning electron micrographs (SEM) of samples with upper bainite (no accelerated cooling) and lower bainite (CR2). The ferrite in lower bainite meets at angles to each other making the structure look needlelike, similar to martensite. Figure 5 shows the upper and lower bainite structures SEM. Even at magnifications  $\sim 10,000$  times it is still difficult to distinguish the individual laths in the lower bainite. It is also evident from Figure 5 that the refined microstructure in the lower bainite sample is the reason for the higher strength of this material.

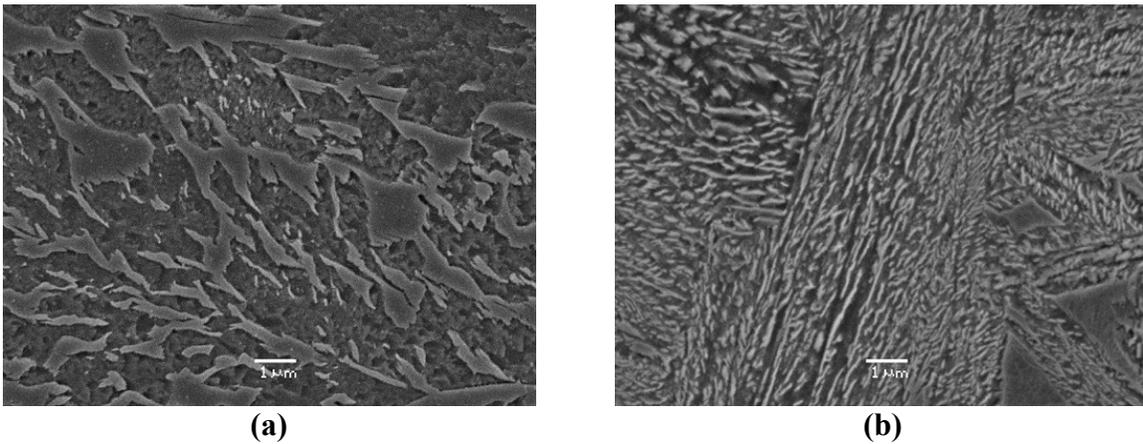
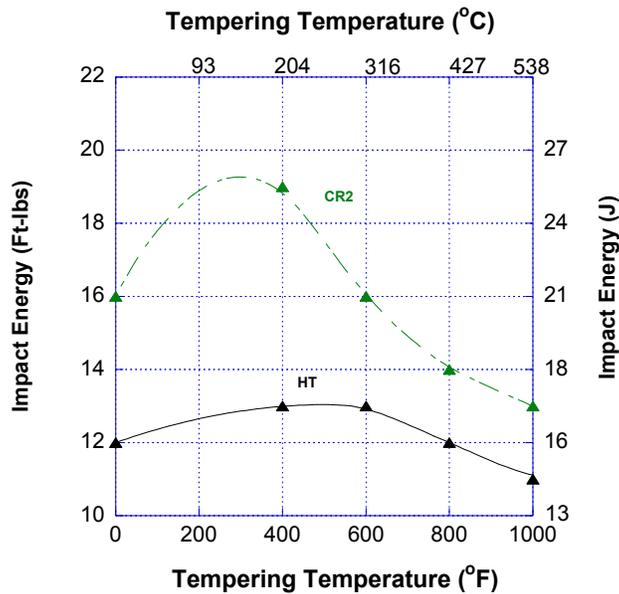
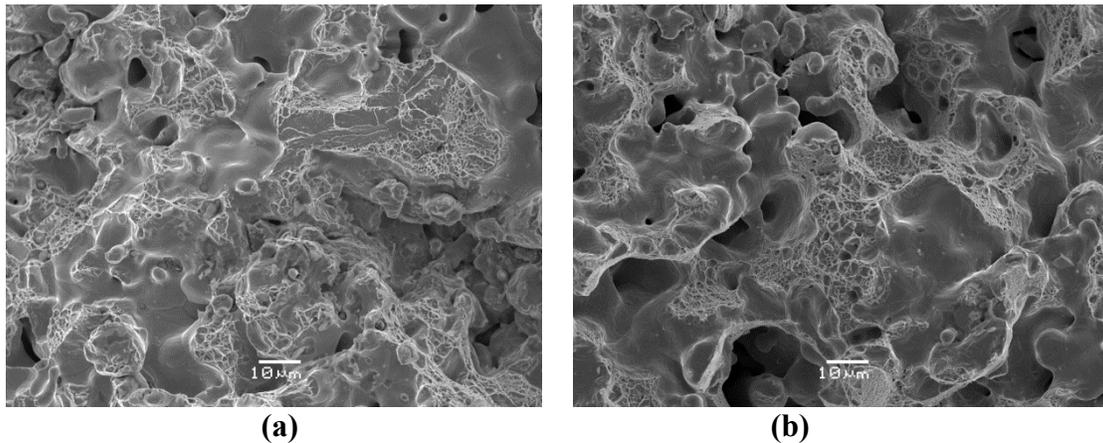


Figure 5: SEM images of: (a) alloy with no cooling- upper bainite and (b) cooling rate (CR2)- lower bainite.

The other significant finding (when looking at the ultimate tensile strength) is that the mixed microstructure (CR2 and CR3) performs nearly the same as the fully martensitic steel that is austenitized and oil quenched. Although the heat treated material has higher ultimate strength as quenched, the material cannot be used in this condition as it is extremely brittle and is prone to quench cracking. When the three materials are tempered at temperatures ranging from 204 to 316 °C (400 to 600 °F), the mixed microstructures of lower bainite and martensite exhibit nearly the same ultimate tensile strength as those of the fully martensitic structures. It has been found that in dual phase materials the mechanical properties are affected by the size, shape and distribution of the second phase, in this case bainite [14-17]. The work of these authors suggests that the lower bainite forming in large volumes, and being fine in nature, actually refines the martensite grain size in the alloy. This finer grain size leads to higher strength. The other advantage of the mixed microstructure with high bainite levels is that it achieves this strength level with a much higher elongation than the martensitic steel (2.2% versus 1.0%).

Another interesting feature of the bainite/martensite mixed microstructure is that, at a tempering temperature of 316 °C (600 °F) the material has the same ultimate tensile strength as the fully martensitic (heat treated) structure but much higher toughness. Figure 6 shows that the mixed microstructure has improved impact energy over the fully

martensitic alloy over a range of tempering temperatures. It has been shown that wrought alloys with a mixed microstructure have higher impact values at both low and high temperatures than the fully martensitic structure [18]. These studies showed that since the lower bainite had a fine microstructure the energy to propagate a crack is similar to that in a fully martensitic structure, but the energy to initiate a crack was much lower in the mixed microstructure due to the softer lower bainite. This appears to be supported by the data shown in Figure 6 as there is an improvement in toughness with the lower bainite/martensite structure.



(c)

Figure 6: Fracture surfaces of: (a) alloy utilizing cooling rate 2 (CR2), and (b) heat treated sample, and (c) corresponding impact energy versus tempering temperature.

In evaluating the fracture surfaces of the tested impact bars, the bainitic sample (Figure 6a) is characterized by a combination of mostly dimples with frequent areas of cleavage and quasi-cleavage. In comparison, the quenched and tempered sample, Figure 6b, exhibited the dimpled surface with rarely encountered small areas showing cleavage. These observations coincide with the findings of Barranco, et al. in their study of the effects of tempering on martensitic and bainitic steels [18]. The presence of the smooth pore surfaces was common to both samples and typical for tested porous PM compacts.

### Economics of Lower Bainite

Although this work was an exploratory study on bainite, some comments are warranted in relation to performance and cost of such an alloy in comparison with those of currently available PM alloy systems. From the results, it is obvious that an alloy with a mixed microstructure of lower bainite and martensite can provide attractive combinations of strength and ductility. Table III compares the CR2 processed material to MPIF diffusion alloy steels in the density range of 6.95 to 7.05 g/cm<sup>3</sup>. The cost savings are difficult to quantify. However the use of accelerated cooling, in combination with proper alloy design, can lead to a high performance material with lower alloy costs. In addition, these alloys will not require a heat treatment, other than tempering and would eliminate oil in the part. These cost savings can be balanced against the use of high temperature sintering. The two MPIF alloys shown in Table III use combinations of copper, molybdenum, and nickel in excess of 3 w/o while the CR2 material utilizes lower amounts of less expensive alloy elements such as chromium, manganese and silicon. Although not the purpose of this study, it would seem reasonable that an alloy could be designed to utilize low cost alloying elements in combination with accelerated cooling to produce a lean alloy that would approach the properties of a quench and tempered steel. Since the sintering temperature of PM steels is above the austenitizing temperature it would seem logical that an alloy with sufficient hardenability could be designed to utilize sintering conventional sintering temperatures of around 1120 °C (2050 °F).

Table III. Comparison of Mechanical Properties of Experimental Alloy with MPIF Heat Treated Grades (Density = 6.95-7.5 g/cm<sup>3</sup>).

Material Designation Code	TRS	TRS	Impact Energy		Apparent Hardness	UTS		0.20% Offset Yield		Total Elongation
	(10 <sup>3</sup> psi)	(MPa)	(J)	(ft.lbf)	(HRC)	(10 <sup>3</sup> psi)	(MPa)	(10 <sup>3</sup> psi)	(MPa)	(%)
FD-0205-120HT	190	1307	9	7	33	115.0	791	D	D	<1
FD-0405-130HT	200	1376	9	7	35	140.0	963	D	D	<1
Experimental Alloy (CR2)	287	1975	22	16	23	148.0	1018	113.5	781	2.2

## CONCLUSIONS

- The chemistry of a low alloy PM steel was designed successfully to give high levels of lower bainite in a conventional sintering furnace utilizing accelerated cooling.
- The tensile properties of this alloy, when tempered in the range of 200-315 °C (400-600 °F) led to ultimate tensile strengths close to the heat treated properties of the same alloy.
- The impact energy of the alloy with high levels of lower bainite was superior to the same alloy with a heat treated martensitic structure.
- The use of accelerating cooling with proper alloy design, in some cases, may eliminate the need for heat treating and therefore eliminate the oil on parts.
- The potential exists for alloys to be designed that can be sintered at conventional temperatures (1120 °C (2050 °F) and still exhibit significant amounts of lower bainite. This will lead to alloys with improved properties.
- The use of steels containing lower bainite should lead to leaner alloys systems, thus lowering the overall cost.

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