

PRECIPITATION HARDENING P/M STAINLESS STEELS

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ABSTRACT

Applications requiring high strength stainless steels are growing rapidly. Precipitation-hardening stainless steels have seen limited use in powder metallurgy despite their high strength. Strengthening of these alloys is achieved by adding elements such as copper and niobium, which form intermetallic precipitates during aging. The precipitation-hardening grades exhibit corrosion resistance levels comparable with those of the chromium-nickel (300 series) grades. The physical properties and microstructures of two precipitation hardening PM stainless powders are presented: 17-4 PH, a high-chromium, martensitic precipitation hardening stainless steel, has been optimized for use in PM applications; and a new low chromium (12 w/o) alloy that utilizes copper in the precipitation reaction. This alloy (410LCu), is considered to be a cost-effective alternative in applications that require high strength and moderate corrosion resistance.

INTRODUCTION

Precipitation hardening (commonly called age-hardening) occurs when, two phases precipitate from a supersaturated solid solution.¹ For example:

Precipitation: Solid A \rightarrow Solid A' + Solid B. (1)

The primary requirement of an alloy for precipitation hardening is that the solubility of B in A decreases with decreasing temperature, so that a supersaturated solid solution forms on rapid cooling.

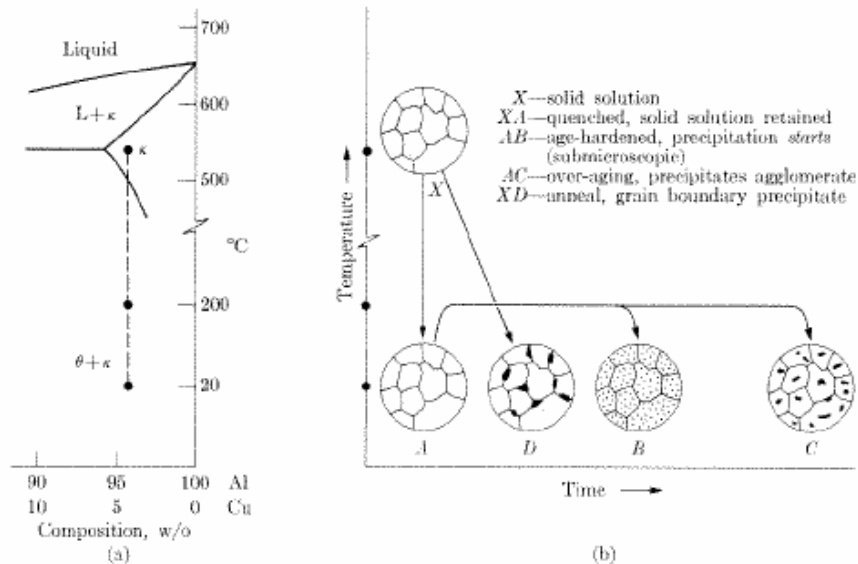


Figure 1: Precipitation hardening sequence.¹

Strengthening as a result of precipitation hardening takes place in three steps²:

- (1) Solution treatment, in which the alloy is heated to a relatively high temperature that allows any precipitates or alloying elements to go into a super-saturated solid solution. Typical solution treatment temperatures are in the range of 982 °C to 1066 °C (1800 °F to 1950 °F).
- (2) Quenching, in which the solution treated alloy is cooled to create a super-saturated solid solution. The cooling can be achieved using air, water or oil. In general, the faster the cooling rate the finer the grain size which can lead to improved mechanical properties. Regardless of the method of cooling, the cooling rate must be sufficiently rapid to create a super-saturated solution.
- (3) Precipitation or age hardening, in which the quenched alloy is heated to an intermediate temperature and held for a period of time. At these intermediate temperatures the super-saturated solid solution decomposes and the alloying elements form small precipitate clusters. The precipitates hinder the movement of dislocations and consequently the metal resists deformation and becomes harder.

Austenitic, semi-austenitic and martensitic precipitation hardening stainless steels currently exist. The austenitic and semi-austenitic compositions were originally designed for aerospace applications involving high temperatures (>704 °C (1300 °F)) where high strength is required. The martensitic precipitation-hardenable alloys are more widely used than the austenitic and semi-austenitic grades, and are the focus of this paper.

BACKGROUND

Two alloys were examined in this study. The first alloy, 17-4 PH (UNS #S17400), is a chromium-nickel-copper precipitation hardening alloy used for applications in the aerospace, chemical, petrochemical and food processing industries. Common wrought products made from 17-4 PH stainless steel include valves, fittings, springs, fasteners and boat shafts. 17-4 PH powder has been widely studied for PM injection molding applications.³⁻⁵

The second alloy examined was a variation of UNS J91151, primarily a casting grade alloy. In the PM version studied in this paper, the composition was adjusted to allow for precipitation hardening via copper. This alloy also contains a unique balance of elements that strengthen the martensite matrix, while providing a high level of ductility. UNS J91151 finds

applications in pump impellers and casings due to its resistance to cavitation; this property is typically a function of the hardness of the alloy.⁶ The chemical compositions of the two stainless steel grades are shown in Table I.

Table I: Composition of Martensitic Precipitation Hardening Stainless Steels (w/o).

Alloy UNS No.	C	S	P	Si	Cr	Ni	Cu	Mn	Nb + Ta
S17400	0.07	.030	.040	1.0	15.0	3.0	3.0	1.0	.15
	Max.	Max.	Max.	Max.	17.0	5.0	5.0	Max.	.45
J91151	0.15	.030	.040	1.5	11.5	1.0	3.0	1.0	---
	Max.	Max.	Max.	Max.	14.0	Max.	5.0	Max.	---

ALLOY PREPARATION AND TESTING

The powders used in this study were produced by water atomization with a typical particle size $<150\ \mu\text{m}$ (-100 mesh) and with 38 to 48 w/o $<45\ \mu\text{m}$ (-325 mesh). All the alloying elements were prealloyed into the melt prior to atomization.

The prealloyed powders were mixed with 0.75w/o Acrawax C lubricant. Samples for transverse rupture (TR) and tensile testing were compacted uniaxially at 690 MPa (50 tsi). All the test pieces were sintered in a high temperature Abbott continuous-belt furnace at 1260 °C (2300 °F) for 45 min in hydrogen with a dewpoint of $-40\ \text{°C}$ ($-40\ \text{°F}$), unless otherwise noted.

Prior to mechanical testing, green and sintered density, dimensional change (DC), and apparent hardness, were determined on the tensile and TR samples. Five tensile specimens and five TR specimens were tested for each composition. The densities of the green and sintered steels were determined in accordance with MPIF Standard 42, while tensile testing followed MPIF Standard 10. Impact energy specimens were tested in accordance with MPIF Standard 40. Apparent hardness measurements were conducted on tensile, TR and impact specimens, following MPIF Standard 43.

Rotating bending fatigue (RBF) specimens were machined from test blanks that were pressed at 690 MPa (50 tsi) and sintered at 1260 °C (2300 °F). The dimensions of the test blanks were 12.7 mm x 12.7 mm x 100 mm. RBF tests were performed using rotational speeds in the range of 7,000-8000 rpm at $R = -1$ using four fatigue machines simultaneously. Thirty specimens were tested for each alloy composition, utilizing the staircase method to determine the 50% survival limit and the 90% survival limit for 10^7 cycles (MPIF Standard 56).

Metallographic specimens of the test materials were examined by optical microscopy in the polished and etched conditions. Etched specimens were used for microindentation hardness testing, per MPIF Standard 51.

Salt spray testing on TR bars was performed according to ASTM Standard B 117-03. Five TR bars per alloy (prepared as previously described) were tested. The percent area of the bars covered by red rust was recorded as a function of time. The level of corrosion was documented photographically.

RESULTS AND DISCUSSION

ALLOY DEVELOPMENT: 17-4 PH

The physical properties and processing conditions of 17-4 PH produced by conventional press and sinter PM techniques are not well documented. Work by Reinshagen et al⁷ indicate that 17-4 PH has not seen wide usage due to low powder compressibility. These authors also showed that with an increase in the -325 mesh fraction, the atomized powder would sinter to higher densities and negate the lower compressibility. However, with the advent of new stainless steel processing routes,⁸ it is now possible to produce a powder with both low carbon and nitrogen levels that can be compacted to reasonable green densities. Simultaneously, through a new high performance atomizing process, a finer sieve distribution, results in a higher sintered density. The use of high temperature sintering is also useful when processing 17-4 PH.

Table II gives the composition of the 17-4 PH used in this study. The notation “CA” refers to material produced by conventional atomization and the notation “HPA” refers to material produced using the new high performance atomization process.

Table II: Composition of PM 17-4 PH Stainless Steel (w/o)

Alloy	C	S	O	N	P	Si	Cr	Ni	Cu	Mn	Mo	Cb
17-4PH CA	0.023	0.006	0.25	0.020	0.008	0.78	17.43	4.67	3.84	0.06	0.03	0.25
17-4PH HPA	0.011	0.005	0.23	0.018	0.009	0.86	17.34	4.61	3.92	0.20	0.02	0.26

The advanced high-performance processing route for stainless steel powders utilizes an argon oxygen decarburization unit that removes both carbon and nitrogen from the melt. The most common method for producing stainless steel powders, induction melting, does not provide a means to control the carbon and nitrogen levels.

It is well known that both carbon and nitrogen have a negative impact on green density. The green density of 17-4 PH is compared with conventional stainless steels 304L and 434L in Figure 2. Over the range of compaction pressures shown in Figure 2, it can be seen that, while the green density is generally lower than other highly alloyed stainless steels, 17-4 PH exhibits reasonable compressibility.

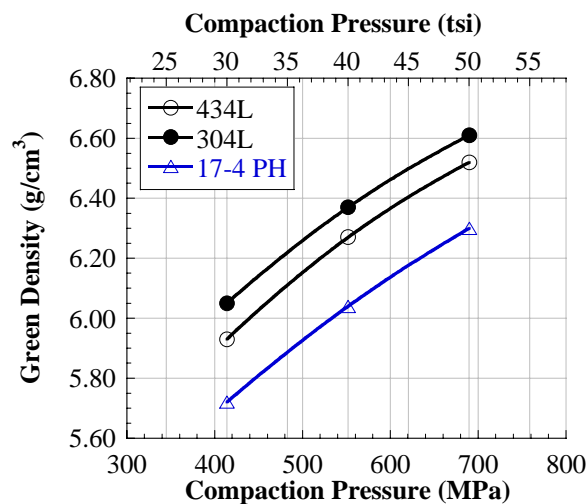


Figure 2. Comparison of green density of 17-4 PH and conventional stainless steels.

In general, lower green densities translate into lower sintered densities. It has been shown by other authors⁷ that sintering of 17-4 PH is enhanced by higher temperatures, and by pure hydrogen atmospheres. The sintered density can also be improved by increasing the minus 325-mesh fraction of the powder or by sintering for longer times. Past studies⁷ have focused on increasing the minus 325-mesh fraction in the powder distribution. Using the HPA route, a material was atomized in which the total distribution was finer including a minus 325-mesh fraction of 44 w/o. This powder and the powder with the standard distribution described earlier, were sintered in a 100% hydrogen atmosphere at 1260 °C (2300 °F) for 20,35, 40 and 60 minutes, as shown in Figure 3.

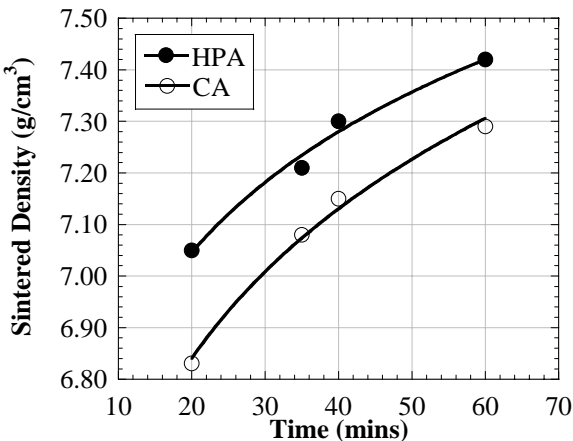


Figure 3: Sintered density of HPA and CA atomized powders as a function of time at temperature.

Results show that even with moderate times at temperature (20 minutes) sintered densities near 7.0 g/cm³ can be achieved. As the time at temperature is increased the sintered density increases and reached 7.40 g/cm³ with 60 minutes at 1260 °C (2300 °F). The material produced from the high-performance atomizing route leads to a finer powder and higher sintered densities. The corresponding mechanical properties are increased, compared with materials produced by conventional atomization. Figure 4 shows the transverse rupture strength and apparent hardness of the HPA powder, over a range of sintered densities. As expected, the mechanical properties increase with increasing sintered density.

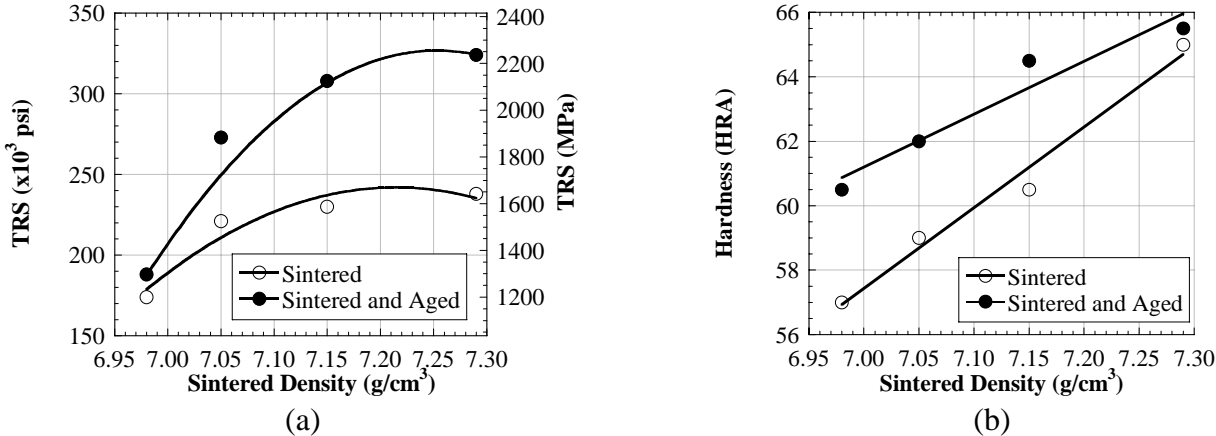


Figure 4. Properties of HPA 17-4 PH as a function of sintered density in sintered and sintered and aged conditions: (a) transverse rupture strength and (b) apparent hardness.

The effect of aging can also be seen in Figure 4. Mechanical property data for wrought steels indicate that precipitation hardening can increase the mechanical properties by as much as 15% to 20%. Although the thermal profile of the PM materials is slightly different to that of the wrought material, a similar response is predicted. The wrought grades of precipitation-hardening alloys are aged at temperatures from 482 °C to 621 °C (900 °F to 1150 °F) for time periods ranging from one to four hours. In order to optimize the mechanical properties of PM 17-4 PH, test coupons were aged at various temperatures and times in a nitrogen atmosphere. The affects of this aging treatment are illustrated in Figure 5. It is seen that the optimum aging temperature for maximum strength and apparent hardness in this alloy occur at about 538 °C (1000 °F) for one hour. Higher temperatures led to over aging (coarser precipitates) and lower mechanical properties. Similar to the wrought grades, mechanical properties increased from 15 to 20%. The strength and apparent hardness values for material aged at 538 °C (1000 °F) for one hour are given in Table III.

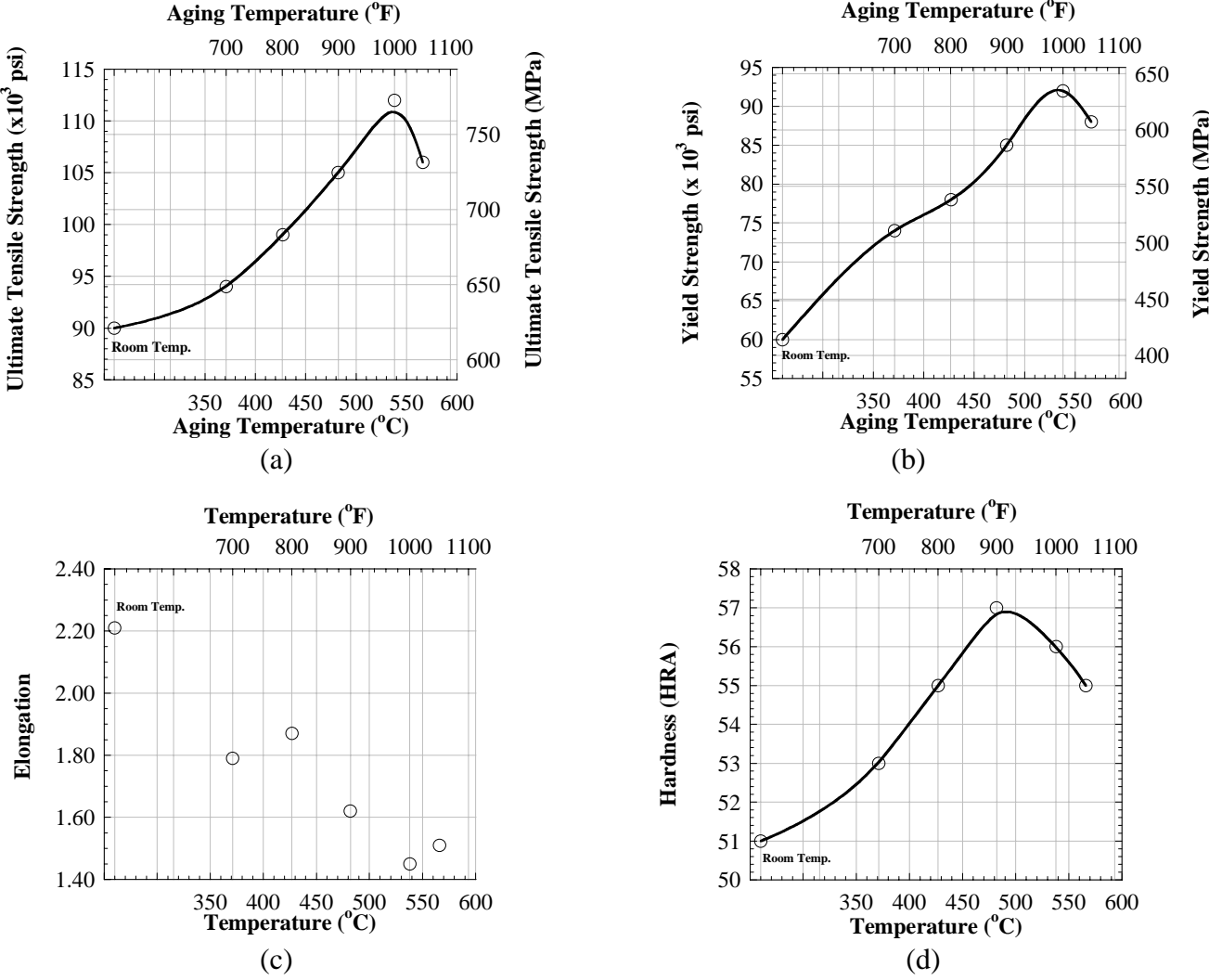


Figure 5: Mechanical properties of 17-4 PH as a function of aging temperature.

17-4 PH is widely used in environments where a level of corrosion resistance comparable to that of the austenitic grades is needed, but in applications that require higher strength and hardness than the austenitic grades can provide. A more realistic comparison of mechanical properties and corrosion resistance would be provided by a duplex stainless steel (22 w/o chromium, 5.5 w/o nickel, 3.5 w/o molybdenum), which has excellent toughness and strength.

Typically, duplex stainless steels are used in applications where the strength of the austenitic stainless steel is inadequate. The high levels of chromium, nickel and molybdenum account for the excellent corrosion resistance of the duplex alloy. The mechanical properties of these three PM alloys are compared in Table III.

Table III: Mechanical Properties of Corrosion Resistant PM Stainless Steels

Alloy	Impact Energy		Apparent Hardness (HRA)	UTS		0.20% OFFSET		Elongation (%)
	(J)	(ft.lbf)		(10^3 psi)	(MPa)	(10^3 psi)	(MPa)	
304L	42	31	36	52	358	31	213	14
DUPLEX	120	89	50	84	578	50	344	10.8
17-4PH Aged	26	19	60	119	819	100	688	1.7

These three alloys were compacted at 690 MPa (50 tsi) and sintered at 1260 °C (2300 °F) in 100% hydrogen. Salt spray testing, performed according to ASTM Standard B 117-03, was conducted for 240 hours; the results can be seen visually in Figure 6. This test is intended as a general guideline as to the performance of the alloys. All three alloys exhibited similar levels of rust. Since the mechanism for corrosion varies by application, specific testing must be undertaken to ensure the satisfactory performance of the alloy.

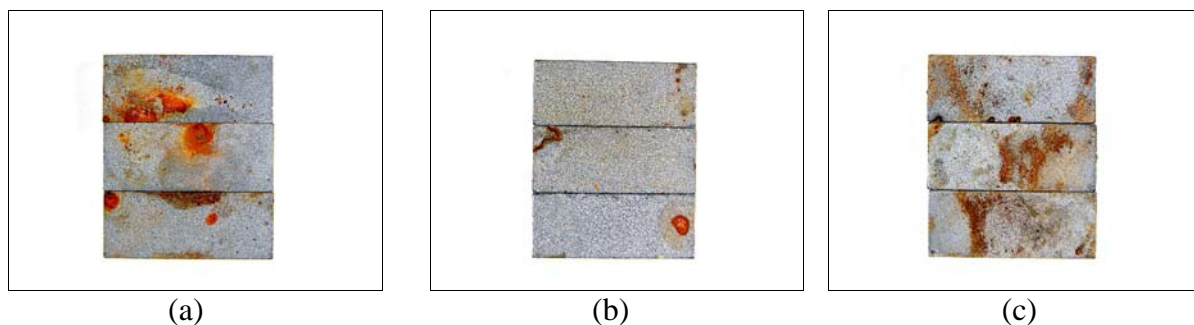


Figure 6. Photographic documentation of salt spray specimens: (a.) 304L, (b.) Duplex, and (c.) 17-4 PH.

ALLOY DEVELOPMENT: 410LCU

There are many applications in which stainless steels with only moderate corrosion resistance (compared to 17-4 PH) but excellent mechanical properties are required. A widely used example is SS-410-90-HT (MPIF designation). This is a PM 410L stainless steel in which graphite is added to the atomized powder and the alloy is sintered in a nitrogen-rich atmosphere. When carbon and nitrogen are added to 410L stainless steel, the microstructure becomes martensitic, thereby increasing both strength and hardness. This grade of stainless steel is used in applications where high strength and hardness are required. The disadvantage of using carbon and nitrogen is that they have a negative impact on corrosion resistance and also reduce impact strength and ductility. Many fabricators request this alloy because there are few alternative compositions. In the casting industry, UNS J91151 (Table I) exhibits many of the properties exhibited by SS-410-90HT. This alloy can be modified by copper to form a martensitic precipitation-hardening stainless steel.⁶

Based on UNS J91151, a series of PM copper containing stainless steel alloys was fabricated. All the powders were produced by water atomization with a typical particle size $<150 \mu\text{m}$ (~ 100 mesh) with 38 to 48 w/o $<45 \mu\text{m}$ (~ 325 mesh). All the alloying elements were prealloyed into the melt prior to atomization. Table IV cites the chemical composition of the precipitation hardening 410LCu alloys.

Table IV: Chemical Composition of 410LCu High-Strength Stainless Steel Grades (w/o).

Alloy UNS J91151	P	Si	Cr	Ni	Cu	Mn	Mo	Cb
410LCu-A	0.011	0.65	12.10	0.08	1.35	0.09	0.01	---
410LCu-B	0.007	0.50	12.81	0.30	2.23	0.06	0.01	
410LCu-C	0.007	0.83	12.65	0.97	2.13	0.05	0.33	
410LCu-D	0.008	0.77	13.00	0.40	1.50	0.05	0.34	---
410LCu-E	0.011	0.71	12.72	0.40	1.48	0.05	0.36	0.36
410LCu-F	0.008	0.73	12.77	1.08	3.06	0.06	0.35	---

The copper content was varied to determine the amount needed for optimum precipitation hardening response. The nickel and molybdenum levels were varied to strengthen and harden the martensite formed in the alloy. Previous studies have shown that there is a syngestic effect between molybdenum, copper and nickel on sintered density.⁹ One alloy was made with the addition of columbium to determine if it would aid in the precipitation reaction.

Compressibility data over a range of compaction pressures show that the addition of copper (Alloy A) reduces green density slightly, when compared with SS-410-90-HT, but is substantially higher than 17-4 PH (Figure 7). Even when nickel and molybdenum are added with increasing levels of copper, the compressibility of the experimental alloys exceeded that of 17-4 PH.

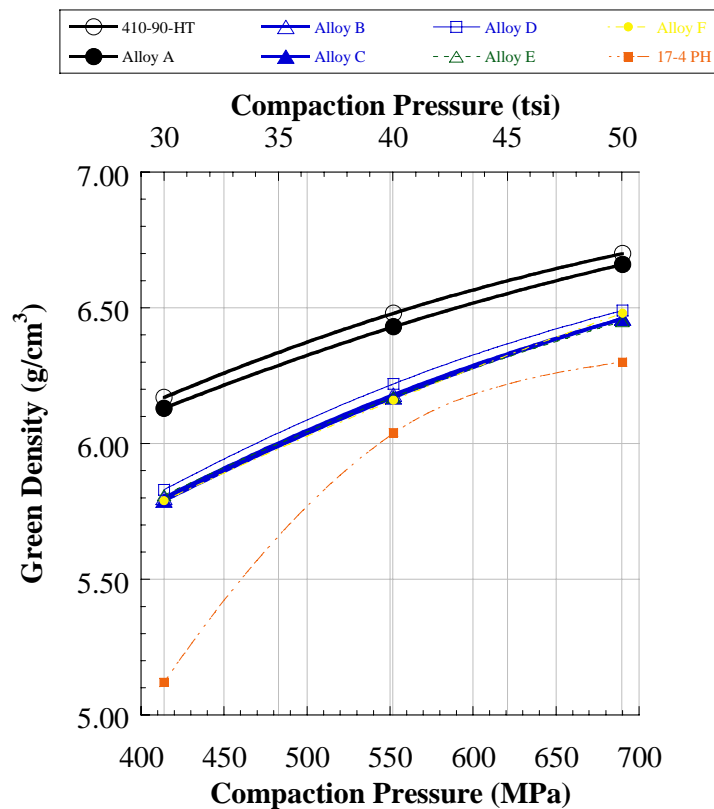


Figure 7. Comparison of green density of experimental alloys.

The static mechanical properties of these alloys are compared in Table V. It is apparent that the alloys with small additions of nickel and molybdenum result in higher strength and hardness. The microindentation hardness of the Alloy A was 214 HV (50 gf) while that of Alloy C (identical copper level but with nickel and molybdenum) was 309 HV (50 gf). The microstructure of Alloy E revealed a fully ferritic structure, which explains the lower mechanical properties of this alloy. Columbium, which was added to aid the precipitation reaction, is also a strong ferrite stabilizer and its addition resulted in a fully ferritic microstructure. Because of their excellent mechanical properties, alloys Alloy C and Alloy F were chosen as the optimum alloys and were subjected to further testing.

Table V: Mechanical Properties of High Strength PM Stainless Steels.

Alloy	TRS		Apparent Hardness (HRB)	UTS		0.20% OFFSET		Elongation (%)
	(10 ³ psi)	(MPa)		(10 ³ psi)	(MPa)	(10 ³ psi)	(MPa)	
410LCu-A	182	1252	84	80	550	57	392	7.5
410LCu-B	186	1280	82	79	544	58	399	5.2
410LCu-C	243	1672	94	105	722	81	557	3.2
410LCu-D	193	1328	84	75	516	55	378	8
410LCu-E	146	1004	75	65	447	59	406	10
410LCu-F	318	2188	102	113	777	87	599	2.8

From a detailed study of aging temperatures and times, similar to the study performed on 17-4 PH, it was concluded that the optimum aging for the 410LCu alloy was one hour at 538 °C (1000 °F). A comparison of as-sintered and aged properties is provided in Table VI. As expected, aging led to improvements in tensile strength and hardness of approximately 15 to 20%. Both alloys also exhibit unusual behavior during the aging process. As the strength and hardness increase during aging, the ductility and impact toughness of the two alloys improves.

Table VI: Sintered and Aged Properties of 410LCu PM Stainless Steels.

Alloy	Impact Energy		Apparent Hardness (HRA)	UTS		0.20% OFFSET		Elongation (%)
	(J)	(ft.lbf)		(10 ³ psi)	(MPa)	(10 ³ psi)	(MPa)	
SS-410L90-HT	39	29	49	92	633	52	358	4.9
410LCu-C Aged	93	69	57	121	832	100	688	5.1
410LCu-F Aged	48	36	60	137	943	116	798	3.7

An examination of the microstructures of these two alloys reveals that both are a mixture of ferrite and martensite. In an earlier paper it was shown that a dual phase microstructure is formed when there is a correct balance of austenite and ferrite stabilizers.⁹ At the normal sintering temperatures for stainless steels (~ 1260 °C (2300 °F)) when the chemistry is balanced properly, the microstructure consists of a mixture of ferrite and austenite. Upon cooling to room temperature, the austenite transforms to martensite and the final microstructure consists of ferrite and austenite. Copper in the alloy should promote the formation of high temperature austenite, which, upon cooling, transforms to martensite. However, as the copper precipitates, it leaves areas in the matrix depleted of copper and it is in those areas that ferrite can form. During aging, the precipitates strengthen and harden the alloys and the ferrite is tempered, improving both ductility and impact toughness, and imparting a unique set of mechanical properties to this alloy.

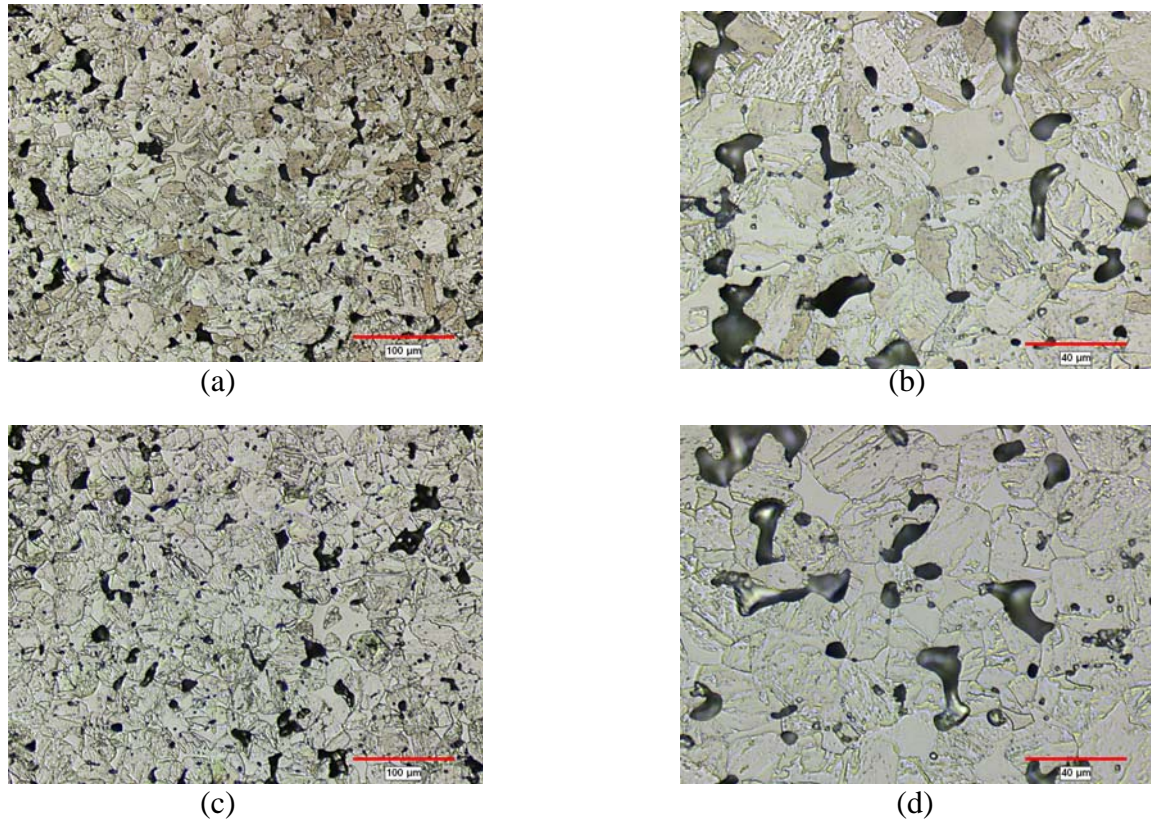


Figure 8: Microstructure of (a) Alloy C (microindentation hardness of martensite 372 HV (50 gf)); (b) Alloy C; (c) Alloy F (microindentation hardness of martensite 250 HV (50 gf)); (d) Alloy F.

The properties of the two new 410LCu alloys are compared with SS410L-90-HT in Table VII. It has been shown that stainless steels with a dual phase microstructure (martensite+ferrite) have higher tensile properties than SS410L-90-HT.⁹ In addition, the ferrite in the microstructure promotes ductility, and precipitation from the copper adds additional strength. In effect, the developmental alloys are superior in mechanical properties because they are dual-phase precipitation hardening alloys.

Table VII: Properties of PM 410LCu versus PM SS410L-90HT

Alloy	Impact Energy		Apparent Hardness (HRA)	UTS		0.20% OFFSET		Elongation (%)
	(J)	(ft.lbf)		(10 ³ psi)	(MPa)	(10 ³ psi)	(MPa)	
SS-410L90-HT	39	29	49	92	633	52	358	4.9
410LCu-C Aged	93	69	57	121	832	100	688	5.1
410LCu-F Aged	48	36	60	137	943	116	798	3.7

In the wrought stainless steel industry, dual-phase-stainless steels were developed as a replacement for weathering steels. The UNS J91151 alloy was developed for moderate corrosion resistance, and the addition of copper was, in general, thought to be beneficial in terms of corrosion resistance. There are numerous applications that are compatible with less-severe corrosive conditions, and, in such cases, ferritic stainless steels are preferred over the more expensive austenitic stainless steels.

The SS410L-90-HT and alloy Alloy C were subjected to salt spray testing using ASTM Standard B 117-03. Figure 9 shows the condition of the test specimens after 240 hours of

exposure. Clearly the 410LCu alloy shows superior performance in this test. Although both alloys exhibit rust (within the first 24 hours), 410LCu appeared to remain unaltered after the initial coating of rust was formed. In contrast, SS-410L-90-HT continued to rust at a constant rate.

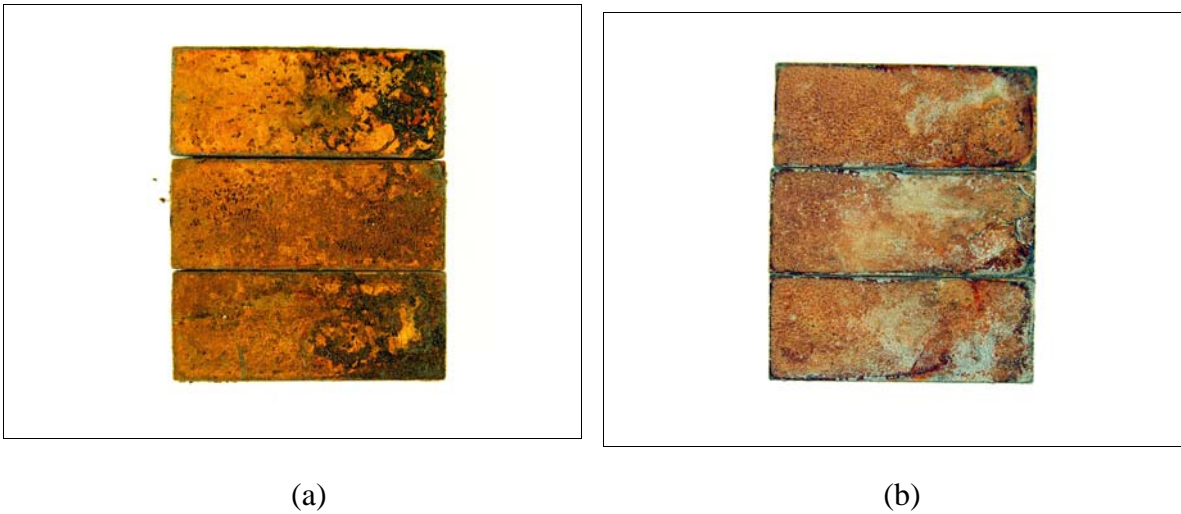


Figure 9. Photographs of TR bars subjected to saltspray testing (a) SS-410L-90HT and (b) Alloy C.

FATIGUE BEHAVIOR OF PM 17-4 PH AND 410LCU ALLOYS

Fatigue tests were performed using the 17-4 PH and the two new 410LCu grades (Alloy C and Alloy F). The results of these tests were compared with other stainless steel fatigue data by Shah et al.¹⁰ in Figure 10. The latter study compared the fatigue strength of various stainless steels as a function of tensile strength.

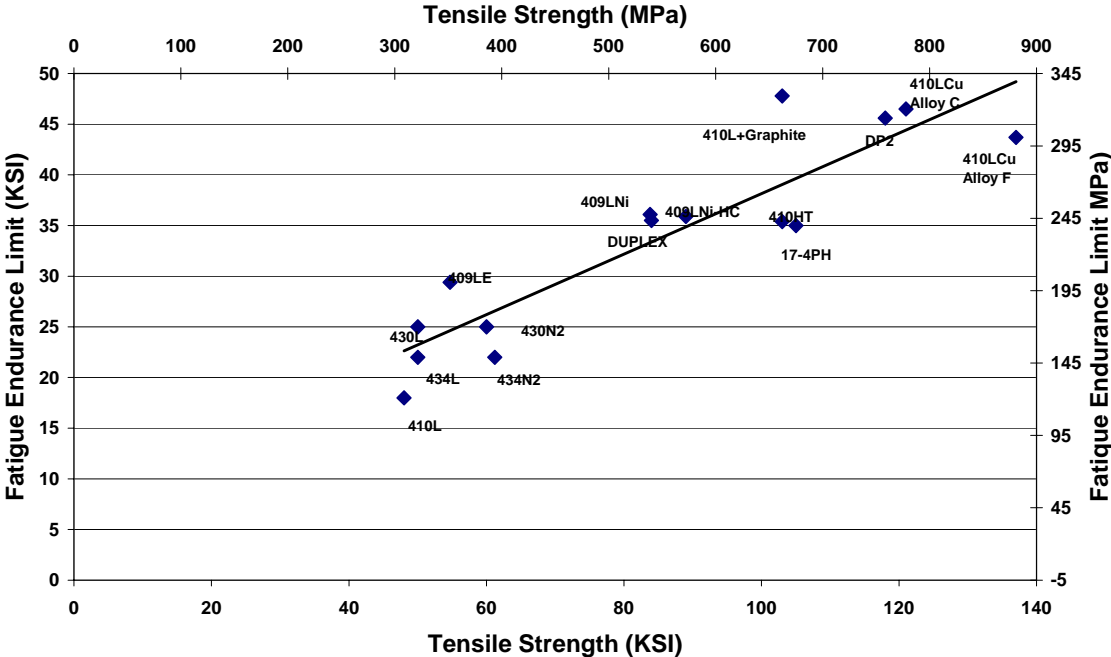


Figure 10. Fatigue endurance limit (for 90% Survival) as a function of tensile strength for various PM stainless alloys.

The 17-4 PH (6.98 g/cm³) exhibited fatigue strength comparable to that of SS-410L-90-HT. With high sintering temperatures, and a finer particle size distribution, it can be expected to out-perform this alloy. The excellent fatigue response of the PM 410LCu alloy appears to be related to its high tensile strength. In general, fatigue crack propagation rates in PM steels are high and the fatigue limit is dictated by crack initiation rather than crack propagation. Thus, resistance to crack initiation increases as the tensile strength increases. Both the 410LCu alloys have high tensile strengths, and therefore higher fatigue endurance limits. It appears that the addition of copper, along with the addition of nickel and molybdenum, leads to harder martensite, which, in this case has a positive effect on fatigue strength.

CONCLUSIONS

- 17-4PH stainless steel parts, which have historically been produced by metal injection molding, can be produced by conventional PM press and sinter operations.
- The mechanical properties of PM 17-4 PH can be improved by utilizing a finer particle size distribution, higher sintering temperatures and longer sintering times.
- The corrosion resistance of PM 17-4 PH is similar to that of 304L, when processed under similar conditions.
- A leaner precipitation hardening grade, PM 410LCu, has been developed for applications that require high strength and toughness, but moderate corrosion resistance.
- The PM 410LCu alloy has a unique combination of high strength, high toughness and fatigue resistance. This is attributed to its' microstructure, which is a dual-phase-precipitation hardened stainless steel.
- PM 410LCu is a cost-effective alloy for applications that require high strength and moderate corrosion resistance, such as pump parts and pump housings.

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