

# THE EFFECT OF SERVICE TEMPERATURE ON THE PROPERTIES OF FERRITIC P/M STAINLESS STEELS

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## ABSTRACT

An area of current development for P/M stainless steel applications is automotive exhaust systems. Recent work in this area has proven the potential application of ferritic stainless steel P/M materials for exhaust flanges to be viable. Lacking in this work, however, is a sufficient database for P/M stainless steels and, in particular, elevated temperature mechanical properties.

A study was undertaken to investigate the effect of service temperature on the mechanical properties of several ferritic P/M stainless steel grades including 410L, 409Cb, 434L and 434Cb. In particular, tensile properties were generated for these alloys and compared with their wrought equivalents. The effect of process conditions and the subsequent microstructure was also considered.

## INTRODUCTION

The increasing acceptance of P/M stainless steel solutions for automotive exhaust applications has focused a major effort on establishing the capability and viability of this technology and the available material options. Thus far, evaluations of standard and enhanced ferritic P/M grades have proven the materials to be more than adequate for most coupling flange and hot exhaust gas oxygen sensor (HEGOS) boss applications. [1] In fact there are several vehicles in mass production today that employ stainless steel exhaust systems featuring P/M 434L flanges and oxygen sensor bosses, including the Ford Contour/Mercury Mystique (since 1996) and the 1997 Chevrolet Corvette. However, the severity of the operating environment, with part temperatures regularly exceeding 1000°F (520°C), the presence of road salt and moisture, and the natural wear factor "by-products" of these conditions -- oxidation and corrosion -- directs one to continue to develop performance data on these P/M grades to gain a thorough understanding of their true capabilities. This report is a continuation of work that has been ongoing for several years in

an attempt to establish baseline mechanical performance data for the commercially available ferritic stainless steel powders when processed into parts and/or test specimens. The grades utilized in this body of work include the standard low carbon materials such as 410L (Fe + 12 w/o Cr) and 434L (Fe + 17 w/o Cr + 1 w/o Mo) -- as well as prealloyed stabilized versions so designated by the Cb suffix: 409Cb (410L + 0.5 w/o Cb) and 434Cb (434L + 0.5 w/o Cb).

The challenges availed by these exhaust applications include those related to the in-service wear environment previously mentioned, along with several secondary operations and assembly difficulties. In an effort to establish sufficient data to afford a capability assessment on several of these areas of concern, a study was designed to learn more about the effect of alloy grade, sintering temperature, sintered density level and the resulting mechanical properties. The effect of columbium, added as a stabilizer to enhance weldability and post-weld corrosion resistance, was also analyzed.

#### MATERIALS AND SAMPLE PREPARATION

Several grades of P/M ferritic stainless steel were chosen to study the effect of processing conditions on mechanical properties at both room temperature and elevated temperatures. The base powder chemical compositions are shown in Table 1. Each base powder was mixed with 1 w/o Acrawax C lubricant prior to compaction.

Base Powder	Cr (w/o)	Mo (w/o)	Cb (w/o)	C (w/o)	O (w/o)	S (w/o)	N (w/o)	P (w/o)	Si (w/o)	Fe (w/o)
Ancor 409Cb	11.2	--	.05	.0015	0.13	0.0007	0.013	0.02	0.80	bal.
Ancor 410L	12.7	--	--	0.018	0.23	0.008	0.021	0.02	0.80	bal.
Ancor 434L	16.0	0.80	--	0.025	0.27	0.008	0.021	0.02	0.80	bal.
Ancor 434Cb	16.2	0.80	0.5	0.021	0.19	0.007	0.020	0.02	0.80	bal.

Table I: Base Powder Chemical Compositions

Test specimens were compacted at pressures ranging from 30 to 50 tsi (415 to 690 MPa) on a hydraulic press in accordance with MPIF standards. Green strength, dimensional change, transverse rupture strength, impact energy and tensile properties were evaluated.

Sintering was performed in a laboratory "batch" furnace at temperatures in the range 2050°F to 2300°F (1120 to 1260°C), mainly in a pure hydrogen atmosphere. A limited part of the study was completed using a 75 v/o H<sub>2</sub> / 25 v/o N<sub>2</sub> atmosphere. The time at sintering temperature was maintained at thirty minutes.

Selected specimens also were prepared for metallographic examination. Optical and advanced microscopy techniques were used where appropriate.

## RESULTS AND DISCUSSION

Green and sintered properties, as a function of density, were developed for 409Cb, 410L and 434L sintered at 2050°F (1120°C) and 2300°F (1260°C) in pure hydrogen.

### GREEN STRENGTH

Figure 1 shows the strong influence of green density on the green strength for all three grades. The 410L and the 434L grades each show similar green strengths for a given density, whereas the 409Cb grade exhibited lower green strength.

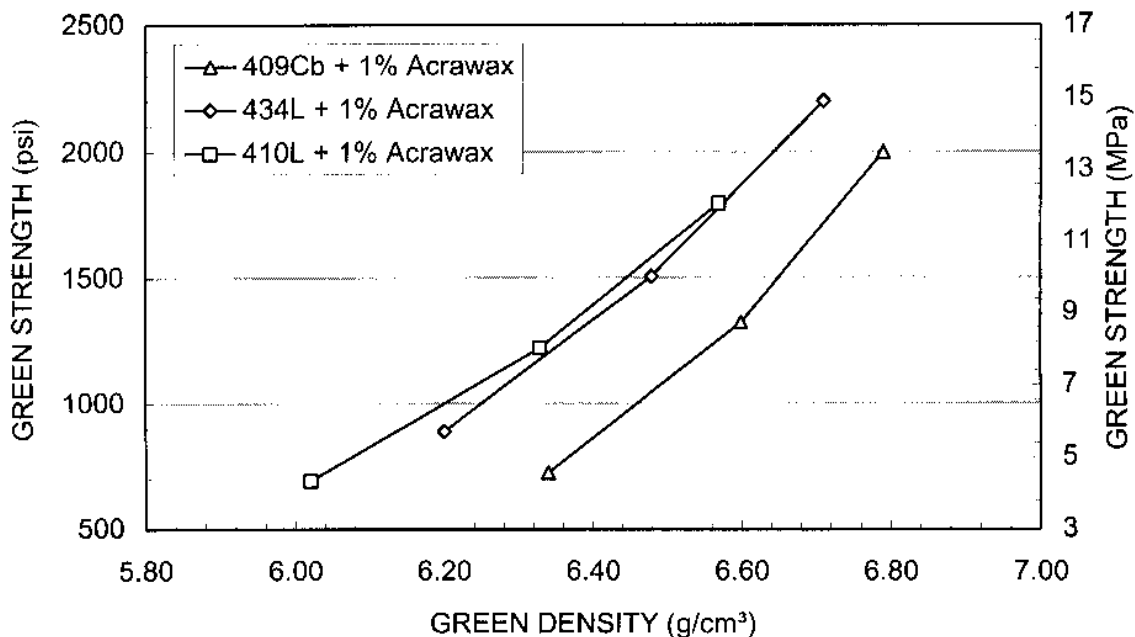


Figure 1: The Effect of Green Density on Green Strength

Recent developments in premix technology have demonstrated the capability of producing green strengths in excess of 5000 psi (35 MPa) in 400-series powder grades. Laboratory data, summarized in Figure 2, shows the green strength for a 434L material comparing a new ANCORMIX™ premix with a traditional lithium stearate premix. The green strength can be increased in excess of 7000 psi (47 MPa) with the use of Ancor® GS-6000 (the first in the ANCORMIX family) versus a maximum level of only 2000 psi (14 MPa) for the conventional premix. [2] The difference in green strength with this new system is consistent across the entire green

density range. This new premix technology offers significant advantages in the processing of P/M stainless steel parts where low green strength remains a challenge.

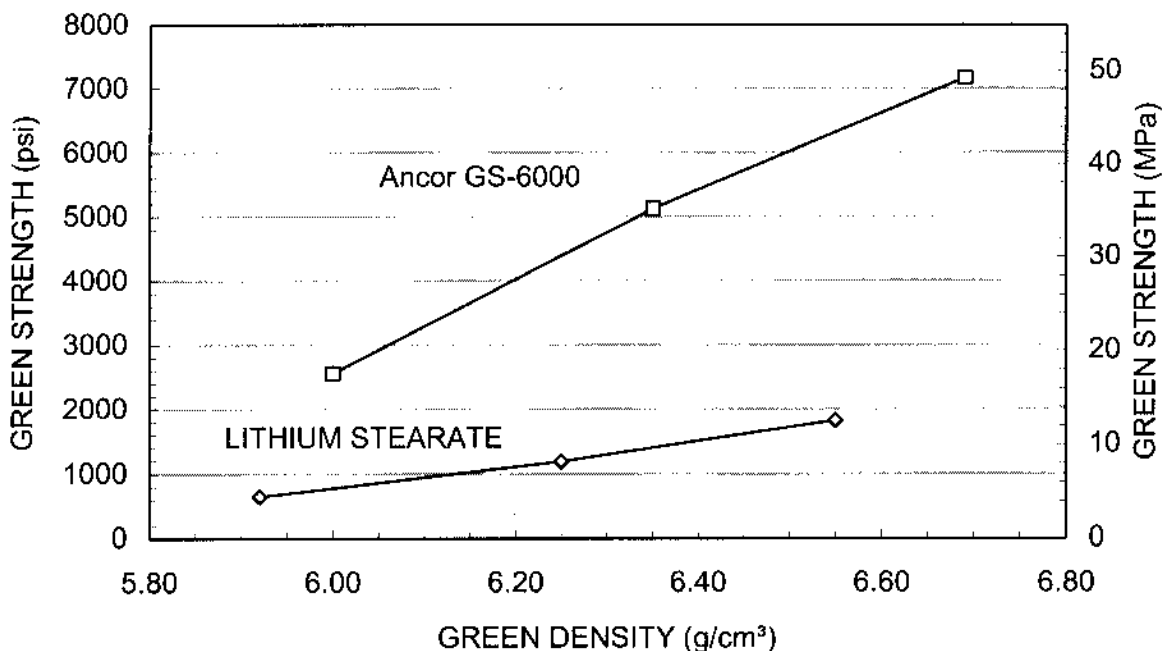


Figure 2: Green Strength Levels for Two 434L Premixes

#### SINTERED PROPERTIES

The influence of high temperature (2300°F/1260°C) sintering can be seen in the sintered properties generated for the three grades. Significantly higher density levels are achieved for a given compaction pressure while mechanical properties are increased dramatically.

In terms of sintered density, the materials sintered at 2300°F(1260°C) show significantly higher density levels than those sintered at 2050°F(1120°C) [Figure 3]. The 409Cb material produced the highest density values, achieving a density of 7.26 g/cm³ at a compaction pressure of 50 tsi (690 MPa). The 434L material had a sintered density of 7.15 g/cm³ and the 410L density was 7.03g/cm³. In general terms, increasing the sintering temperature from 2050°F (1120°C) to 2300°F (1260°C), produced an increase in sintered density of about 0.4g/cm³.

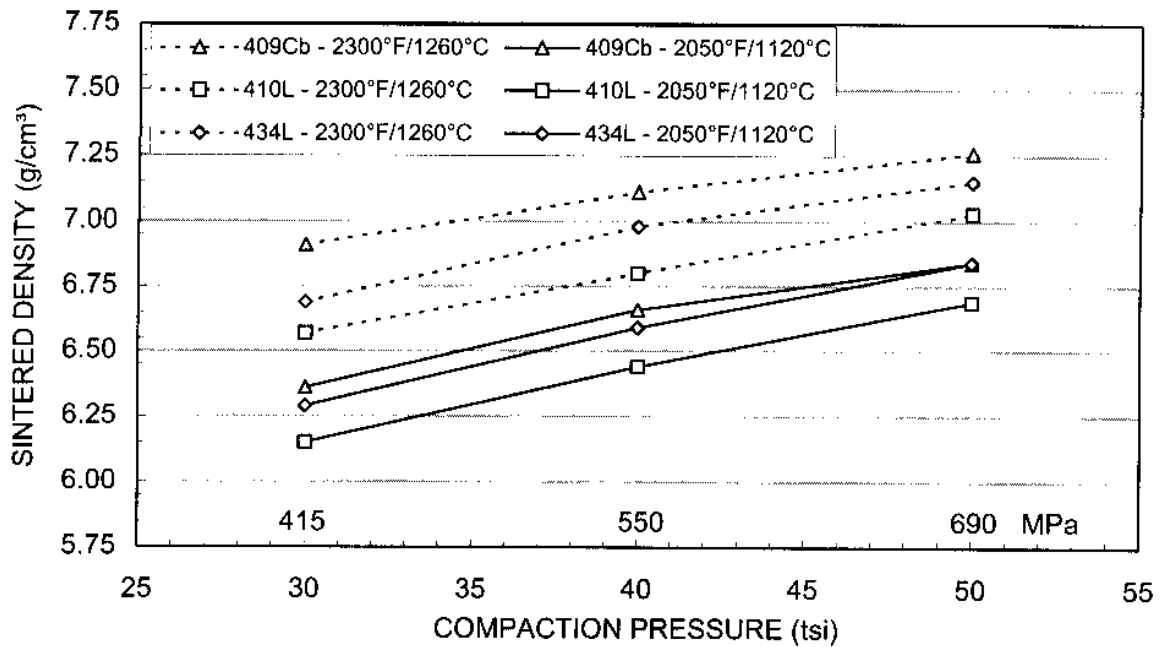


Figure 3: The Effect of Sintering Temperature and Compaction Pressure on Sintered Density of the Three Materials

The dimensional change (from die) results also are evidence that a significant change occurs between the two sintering temperatures (Figure 4). At 2050°F (1120°C) all three grades show between 0.5% and 1.0% shrinkage. Increasing the temperature to 2300°F (1260°C) increases the shrinkage to between 2 and 4%. The 409Cb material exhibited the lowest amount of shrinkage when sintered at 2050°F (1120°C) but shows the largest amount of shrinkage when sintered at 2300°F (1260°C).

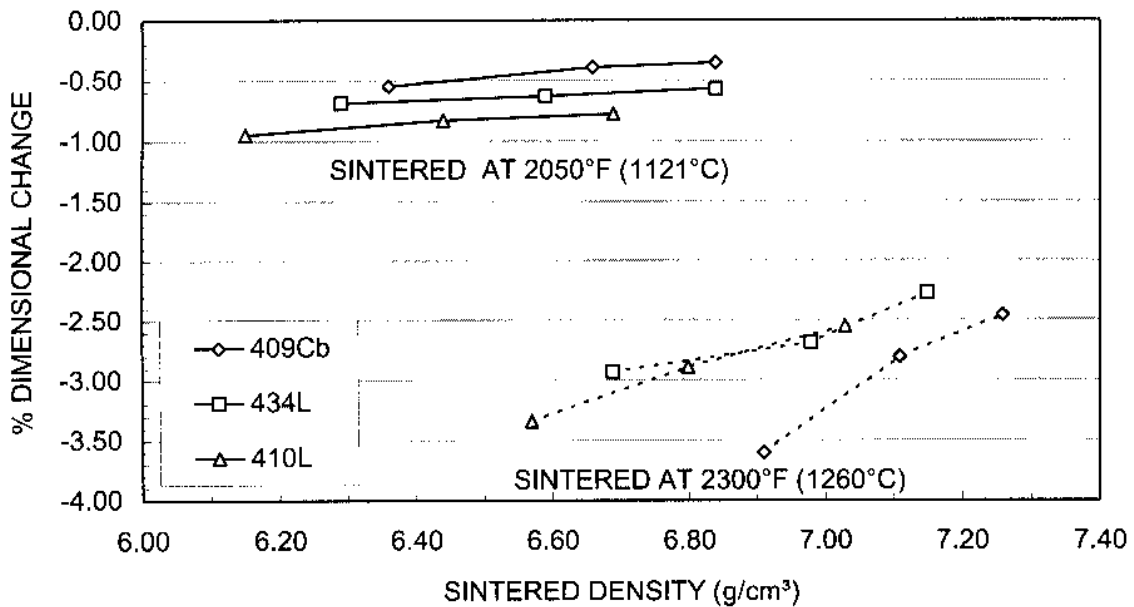


Figure 4: The Effect of Density and Sintering Temperature on Dimensional Change of the Three Materials

Transverse rupture strength data, summarized in Figure 5, show that the 434L material has the highest strength of the three materials for a given density and sintering temperature. It is important to note that when the higher density materials are sintered at 2300°F (1260°C), a significant amount of deflection occurs during testing of these specimens due to the increased ductility of the material. These results are of questionable validity and it is recommended that tensile properties be used to understand the true differences between the materials.

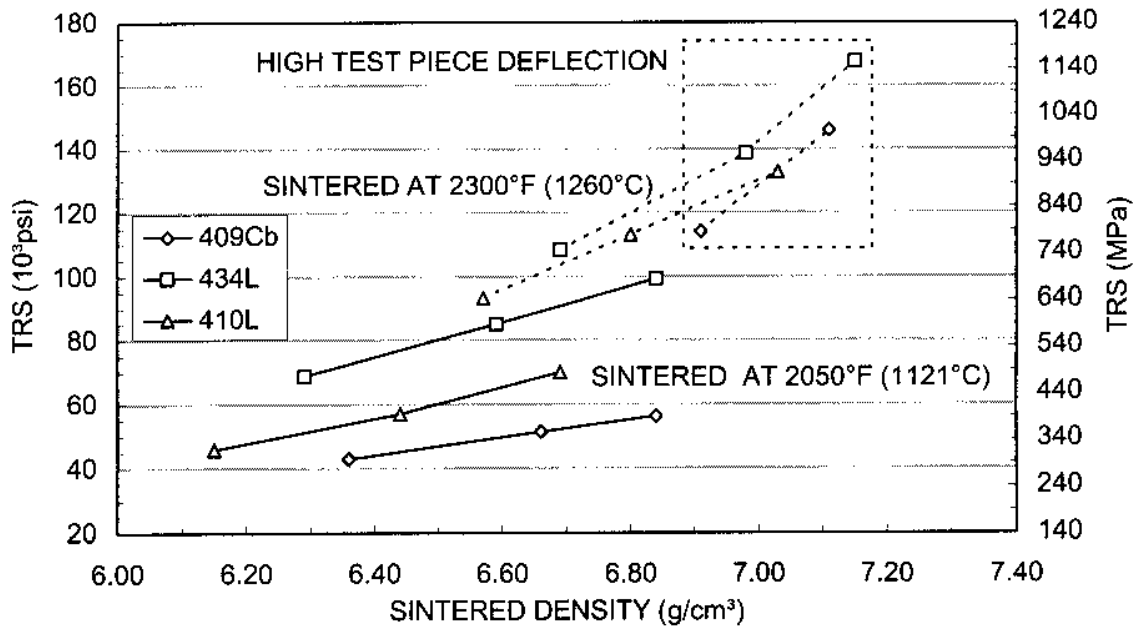


Figure 5: The Effect of Density and Sintering Temperature on the Transverse Rupture Strength of the Three Materials

The ultimate tensile strength results (Figure 6) for each of the three materials indicate a relationship with density. The 434L material achieves the highest tensile strength for a given density and processing condition. The strength gain from sintering the 434L at the higher temperature follows the density increase. The 410L exhibits only slight improvement for a given density level with increased sintering temperature. On the other hand, the 409Cb material showed a significant improvement in strength for a given density level when sintered at 2300°F (1260°C) versus 2050°F (1120°C).

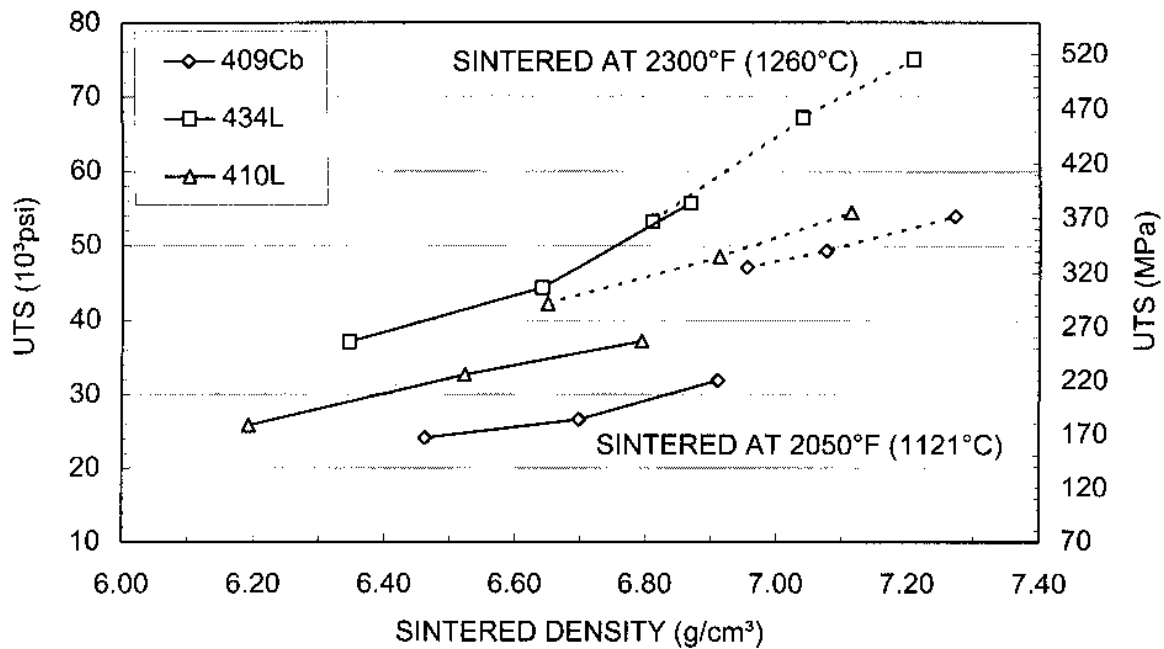


Figure 6: The Effect of Density and Sintering Temperature on the Ultimate Tensile Strength of the Three Materials

Tensile elongation properties (Figure 7) show significant improvement as a result of high temperature sintering. When sintered at 2050°F (1120°C), none of the materials exhibit significant elongation values. In the case of both sintering temperatures, 410L shows higher elongation values than the 434L material. For the 409Cb material, the elongation values are the lowest of the three materials when sintered at 2050°F (1120°C) but result in the single highest value when sintered at 2300°F (1260°C).



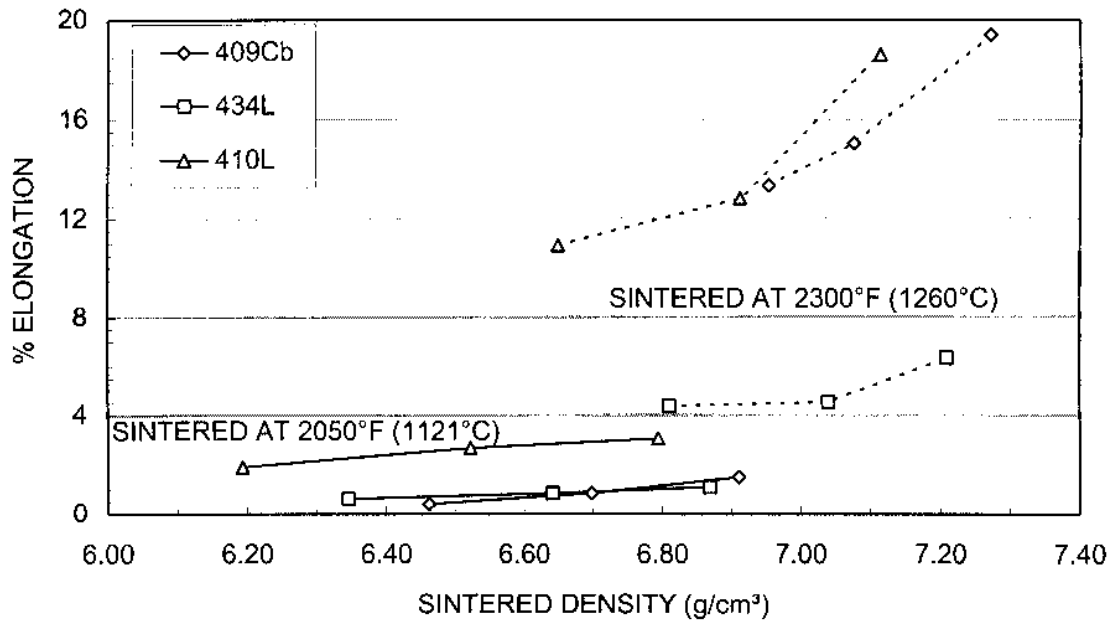


Figure 7: The Effect of Density and Sintering Temperature on the Elongation of the Three Materials

The impact properties (Figure 8) follow nearly an identical trend as found above with elongation. Impact properties are significantly improved by using high temperature sintering. When the three materials are sintered at 2050°F (1120°C), even the effect of density is limited and the best result is less than 20 ft-lbf (27 J). Sintering at 2300°F (1260°C) dramatically improves the impact resistance. The improvement realized is much higher than can be explained by the density increase alone, indicating that the elevated temperature significantly effects inter-particle bonding and pore structure. The 409Cb material shows the strongest reaction to the higher sintering temperature, with impact energies ranging from 42 ft-lbs (57 J) at a density of 7.26 g/cm<sup>3</sup>

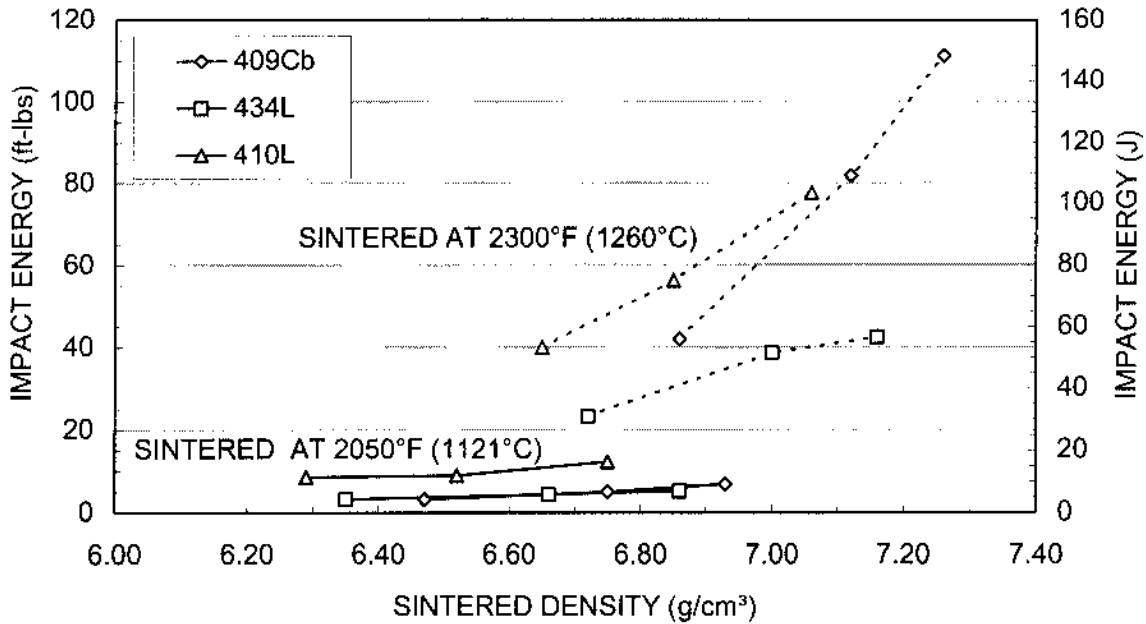


Figure 8: The Effect of Density and Sintering Temperature on the Impact Properties of the Three Materials

The results found during this study are generally as expected. The strength and hardness values were typically higher at a given processing condition for the 434L material than the 410L or the 409Cb materials. Elongation and impact properties are both lower for the 434L material than the other grades tested here. These results can be explained to some degree by the higher alloy content found in the 434L which provides a stronger, less ductile material. Other published information indicates that higher elongation and impact values for 434L can be attained than those generated in this study. [3,4] In all cases, increased sintering temperature leads to improved properties. This is particularly the case with the 409Cb material.

The significant increases seen with the 409Cb material in terms of dimensional change, impact, ultimate tensile strength and elongation, indicate the importance of sintering the columbium bearing materials at elevated temperature to obtain optimal performance. A further detailed study was performed to investigate the processing of Cb bearing materials.

#### COLUMBIUM ADDITIONS TO FERRITIC STAINLESS STEEL

As mentioned above, Cb is added as a stabilizing element to ferritic P/M stainless steels. Stabilizing elements such as Cb and Ti are added to stainless steels to reduce sensitization, typically as a result of a welding operation. A stainless steel material becomes sensitized as a result of the Cr being tied up in the form of Cr-carbides. If the "free" Cr content available to

form an oxide coating is reduced significantly, corrosion resistance also can be reduced. The Cb "ties up" interstitial elements such as carbon and nitrogen during the welding operation, leaving the Cr to form a surface oxide film and provide corrosion resistance. Figure 9 shows a scanning electron photomicrograph of Cb precipitates in a sintered P/M steel. If columbium is to be added to P/M stainless materials, it is important to understand any effects the addition may have on processing or sintered properties.

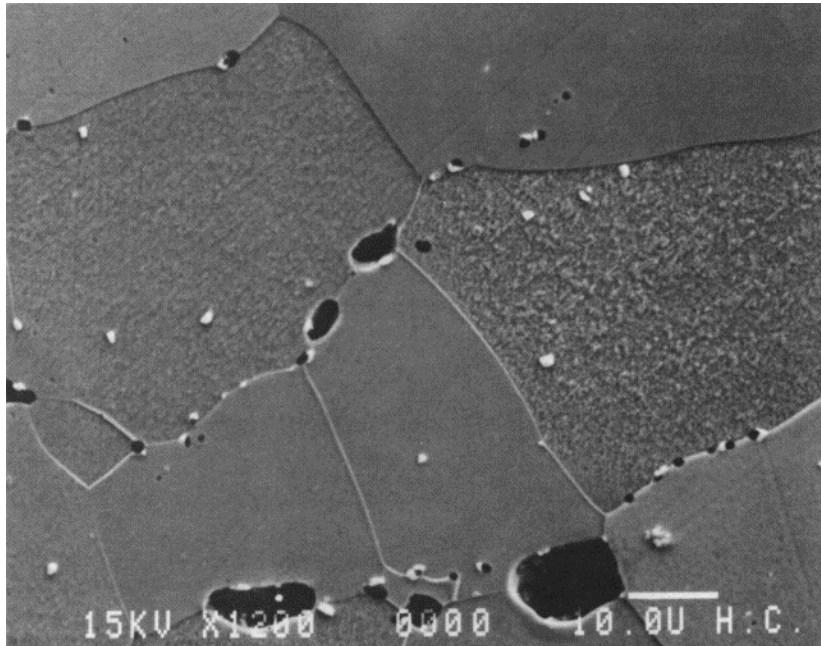


Figure 9: Scanning Electron Photomicrograph of 409Cb showing Presence of Columbium (White Particles) within Grains and at Grain Boundaries (Original Magnification-1200X)

A study was performed where dimensional change, impact energy and ultimate tensile strength were evaluated by comparing 410L with 409Cb and 434L with 434Cb. Test specimens were prepared to the same green density ( $6.6 \text{ g/cm}^3$  for 409Cb/410L and  $6.5 \text{ g/cm}^3$  for 434Cb/434L) and sintered at  $2050^\circ\text{F}$  ( $1120^\circ\text{C}$ ),  $2100^\circ\text{F}$  ( $1150^\circ\text{C}$ ) and  $2300^\circ\text{F}$  ( $1260^\circ\text{C}$ ) in hydrogen for 30 minutes at temperature. Table II lists the results of the testing.

In terms of dimensional change, both of the Cb-bearing materials show less shrinkage when sintered at either  $2050^\circ\text{F}$  ( $1120^\circ\text{C}$ ) or  $2100^\circ\text{F}$  ( $1150^\circ\text{C}$ ) compared with the non-Cb materials.

Interestingly, increasing the temperature to  $2300^\circ\text{F}$  ( $1260^\circ\text{C}$ ) results in higher shrinkage values for the stabilized grades. It appears that at the two lower sintering temperatures, the Cb,

most likely, remains tied to carbon and nitrogen at grain boundaries which causes a reduction in sintering. At 2300°F (1260°C), however, the stabilized grades sinter at an even faster rate than the non-Cb materials.

For the 410L/409Cb comparison, the impact results indicate that sintering temperature has a similar effect as seen for dimensional change. The 410L exhibits higher performance at the two lower sintering temperatures, but at the highest temperature, the 409Cb obtains the best results. For the 434 grades, the stabilized material (434Cb) exhibits better impact properties at all three sintering temperatures.

Material	Sintering Temperature (°F/°C)	Dim. Chg. From Die (%)	Impact Energy (ft-lbs/J)	UTS (10 <sup>3</sup> psi/MPa)
410L	2050/1120	-0.78	12/16	37/256
	2100/1150	-1.21	40/55	48/328
	2300/1260	-2.55	77/105	55/376
409Cb	2050/1120	-0.39	5/7	27/183
	2100/1150	-0.67	17/23	20/206
	2300/1260	-2.81	82/111	49/340
434L	2050/1120	-0.63	4/6	44/306
	2100/1150	-0.88	7/10	51/348
	2300/1260	-2.69	39/52	67/463
434Cb	2050/1120	-0.10	7/10	29/201
	2100/1150	-0.38	17/24	34/234
	2300/1260	-2.81	64/87	53/363

Table II: Sintered Properties of Cb and Non-Cb Bearing Materials

Lower ultimate tensile strengths were seen for both stabilized grades at all three sintering temperatures. The lower strengths may not be unexpected, as columbium would tend to tie up the remaining carbon and nitrogen that would typically help to strengthen the sintered materials. The relative amount of difference is minimized at the 2300°F (1260°C) sintering temperature. This may be in part due to the lower nitrogen levels seen at the higher sintering temperatures (Table III).

Table III also shows an increase in carbon content at 2100°F (1150°C) that may be due to a higher carbon potential in the furnace at the time of sintering. This higher carbon content would tend to strengthen the materials somewhat but degrade ductility measurements such as impact energy. It is also interesting to note that, for a given processing condition, the amount of carbon and nitrogen in the Cb-bearing materials is higher than that found in the non-Cb materials. In the case of nitrogen, the difference is largest at the lowest sintering temperature and becomes much smaller as the sintering temperature increases. This indicates that the control of nitrogen in Cb-

bearing materials is of particular importance.

Grade	Sintering Temperature (°F/°C)	Carbon (w/o)	Nitrogen (w/o)	Oxygen (w/o)
410L	2050°F (1120°C)	0.026	0.004	0.26
	2100°F (1150°C)	0.031	0.004	0.24
	2300°F (1260°C)	0.021	0.001	0.14
409Cb	2050°F (1120°C)	0.032	0.015	0.20
	2100°F (1150°C)	0.069	0.014	0.16
	2300°F (1260°C)	0.020	0.002	0.18
434L	2050°F (1120°C)	0.032	0.012	0.23
	2100°F (1150°C)	0.052	0.009	0.23
	2300°F (1260°C)	0.025	0.002	0.19
434Cb	2050°F (1120°C)	0.038	0.025	0.28
	2100°F (1150°C)	0.056	0.025	0.29
	2300°F (1260°C)	0.028	0.007	0.25

Table III: Chemical Analysis for Stainless Steel Grades Sintered in Hydrogen

Hubbard et al have recommended that for stainless steel P/M components which require weldability, good corrosion resistance, and good mechanical properties, nitrogen and carbon levels should be no higher than 0.03% each [4].

#### ELEVATED TEMPERATURE TENSILE PROPERTIES

Machined round tensile specimens were prepared from the 409Cb, 434Cb and 434L grades following compaction at 50 tsi (690 MPa) and sintering at either 2050°F (1120°C) or 2300°F (1260°C) in pure hydrogen or a 75 v/o H<sub>2</sub> / 25 v/o N<sub>2</sub> gas mixture atmosphere. Tensile testing in air was carried out at room temperature, 1200°F (650°C) and 1600°F (870°C). The results for ultimate tensile strength and elongation are shown in Figures 10 and 11 for materials sintered at 2300°F (1260°C) in hydrogen.

The high sintered strength and low elongation seen previously with the 434L grade is confirmed with the room temperature results from the elevated temperature study. At 1200°F (650°F), the strength falls to about 25 x 10<sup>3</sup> psi from about 80 x 10<sup>3</sup> psi (at room temperature), with a further 15 x 10<sup>3</sup> psi drop at 1600°F (870°C). The two stabilized grades (409Cb and 434Cb) show similar strength to each other at all three test temperatures, whereas the 409Cb material exhibits higher elongation values, especially at 1600°F (870°C).

In order to evaluate the effect of sintering atmosphere and temperature on the elevated temperature tensile properties of

ferritic stainless steels, two additional studies were completed.

The 409Cb material was compacted at 50 tsi (690 MPa) and sintered at 2300°F (1260°C) in both hydrogen and 75 v/o H<sub>2</sub> / 25 v/o N<sub>2</sub> atmospheres. Figures 12 and 13 show the tensile strength and elongation results. The data indicate that sintering atmosphere appears to have a minimal effect on tensile strength, but a major influence on elongation. The presence of nitrogen in the atmosphere may be having two related effects on the resulting properties. The presence of nitrogen leads to the creation of martensite at the grain boundaries of the materials. This can increase the strength considerably but reduce ductility significantly. The presence of this phase also tends to reduce corrosion resistance of the material. The presence of nitrogen also reduces sintering and thus sintered density. As noted above, reduction in density generally leads to deterioration of all mechanical properties.

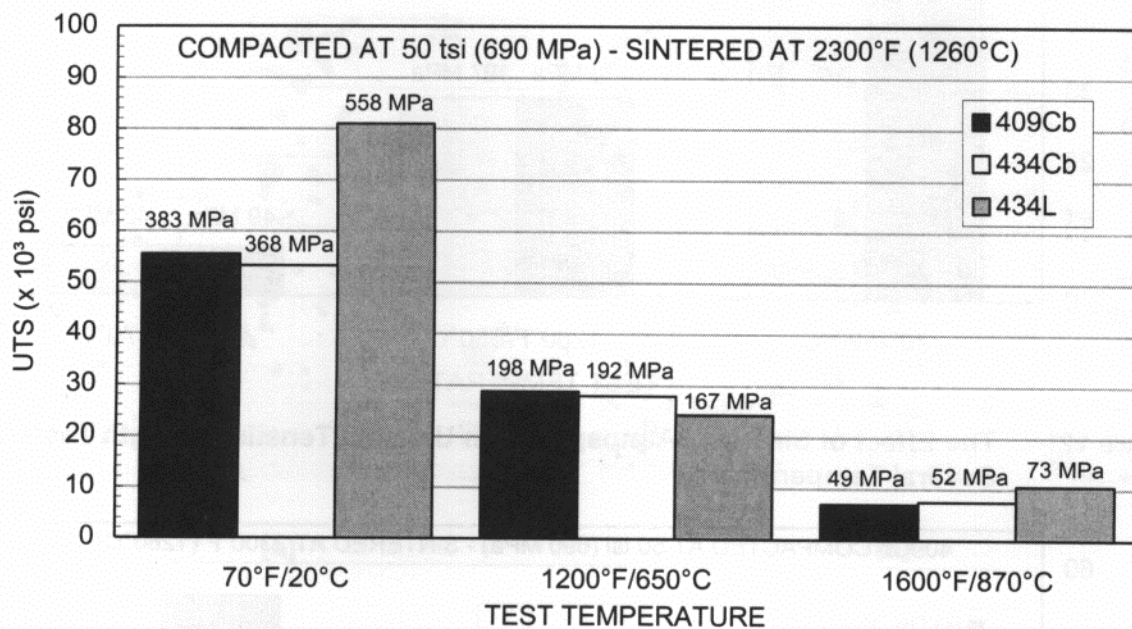


Figure 10: Ultimate Tensile Strength Results from Elevated Temperature Testing of Materials Sintered at 2300°F/1260°C

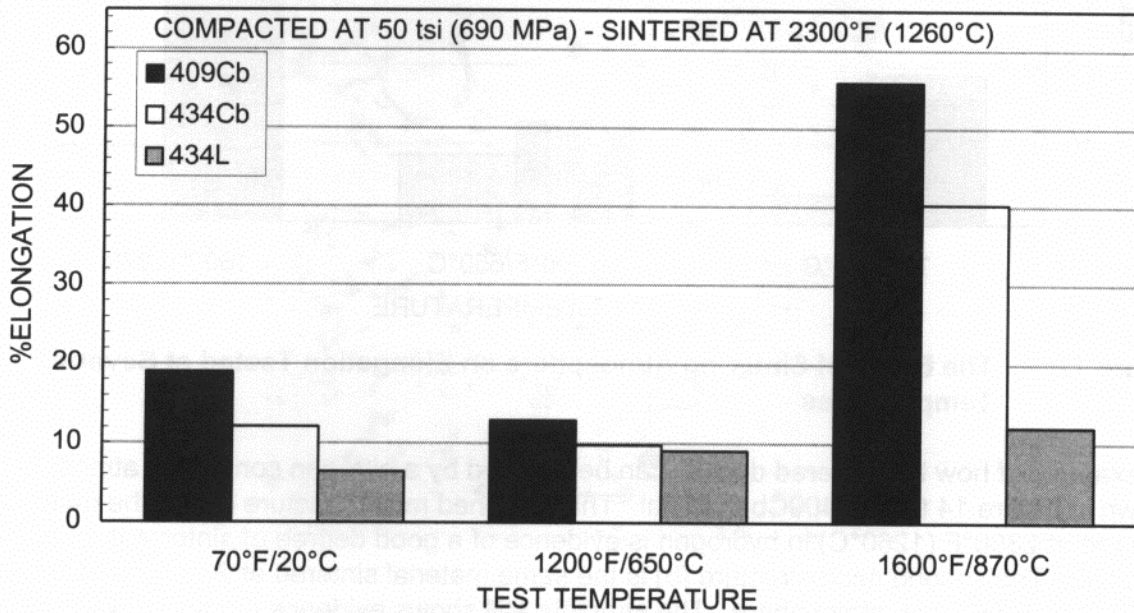


Figure 11: Elongation Results from Elevated Temperature Testing of Materials Sintered at 2300°F/1260°C

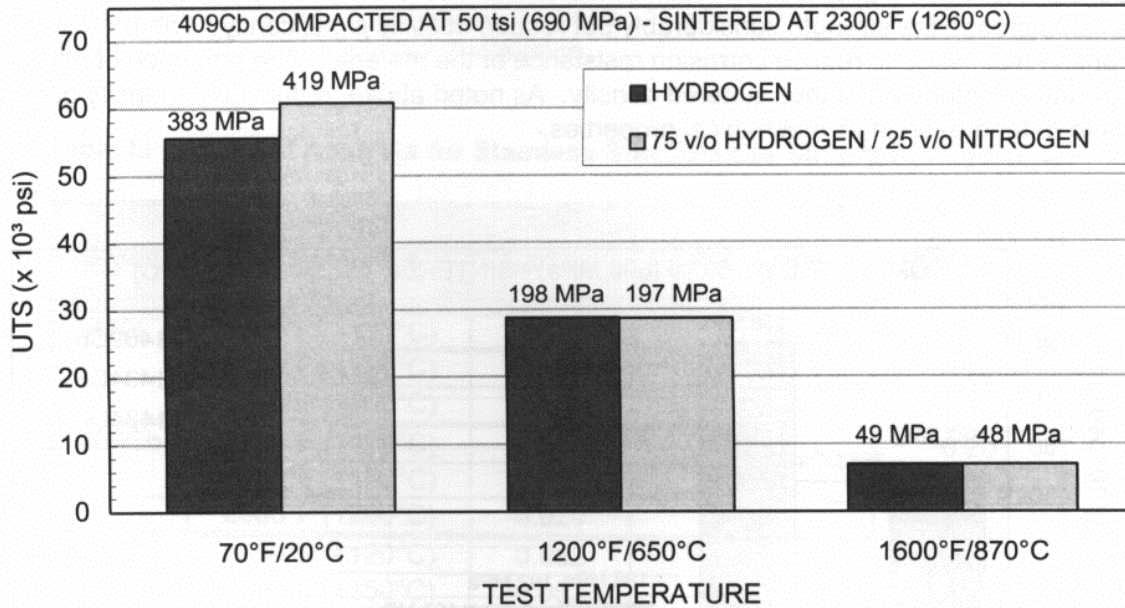


Figure 12: The Effect of Sintering Atmosphere on Ultimate Tensile Strength Tested at Several Temperatures

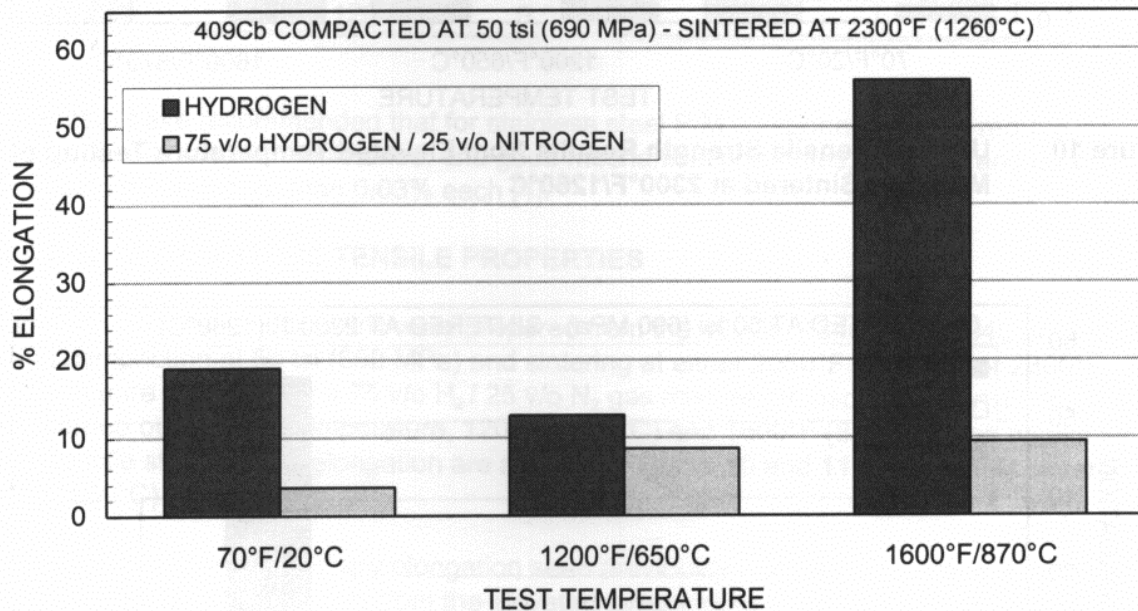
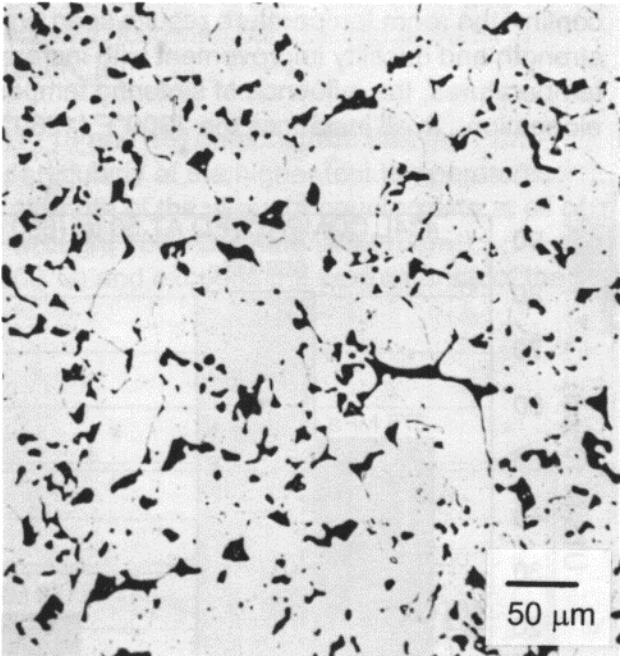
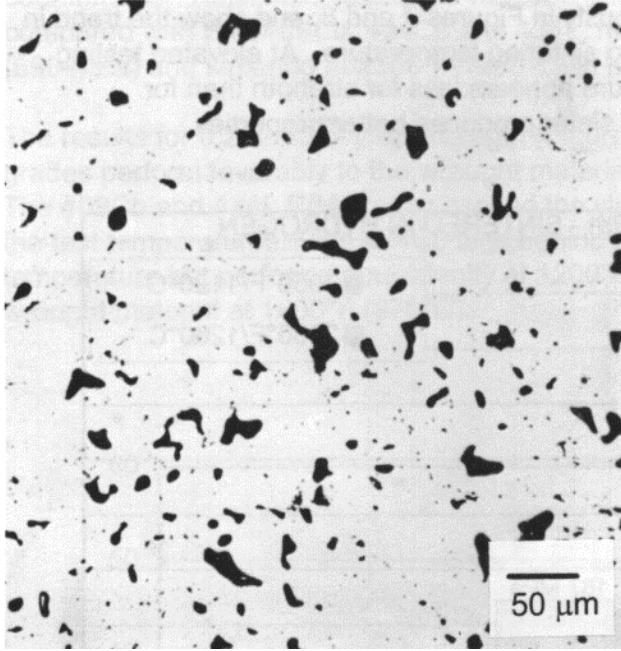


Figure 13: The Effect of Sintering Atmosphere on Elongation Tested at Several Temperatures

An example of how the sintered density can be affected by a nitrogen containing atmosphere is shown in Figure 14 for the 409Cb material. The unetched microstructure (a) for the material sintered at 2300°F (1260°C) in hydrogen is evidence of a good degree of sinter with pore rounding. The second microstructure (b) is the same material sintered at 2300°F (1260°C) but in 75 v/o H<sub>2</sub> / 25 v/o N<sub>2</sub> atmosphere. The structure still shows evidence of prior powder particle boundaries. This structure is very similar to the structure obtained from a 2050°F (1120°C) sinter in hydrogen (c). The negative influence of high interstitial levels of nitrogen and/or carbon in stainless steels has been documented in numerous reports. [4,5] The results generated from this study agree with previous findings.





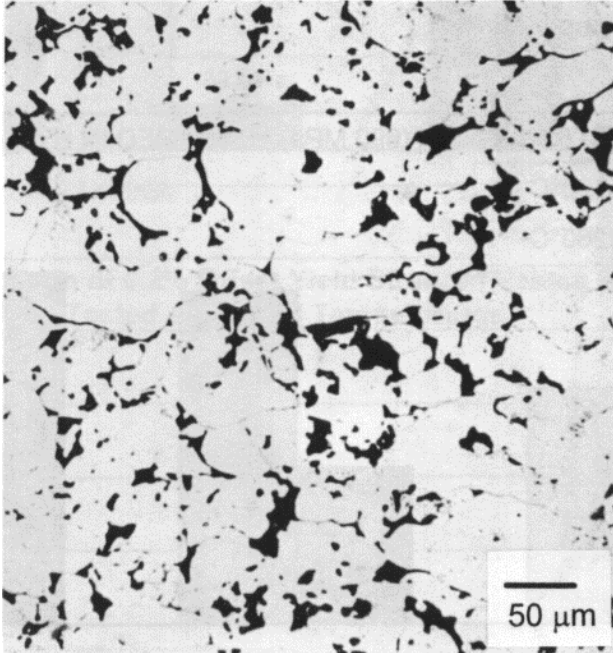


Figure 14: Photomicrographs Indicating the Unetched Sintered Structure of 409Cb Material Sintered in Hydrogen and 75 v/o H<sub>2</sub> / 25 v/o N<sub>2</sub> Atmospheres (Original Magnification 200X)

The influence of sintering temperature on elevated temperature tensile properties was evaluated by comparing 434L sintered in hydrogen at both 2050°F (1120°C) and 2300°F (1260°C). Tensile strength and elongation results are shown in Figures 15 and 16. The data confirm the room temperature results seen previously in Figures 7 and 8, and show the trend in strength and ductility improvement with increasing sintering temperature. At elevated testing temperatures, the influence of sintering temperature appears less for strength than for elongation. In all instances the 2300°F (1260°C) sinter produces better properties.

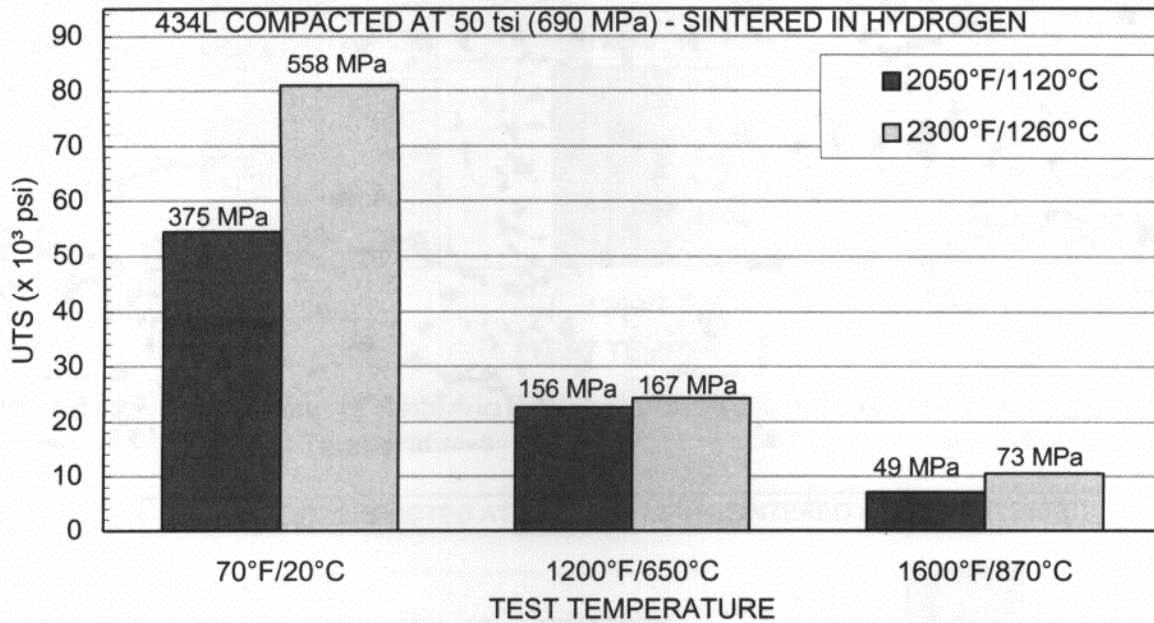


Figure 15: The Effect of Sintering Temperature on Ultimate Tensile Strength Tested at Several Temperatures

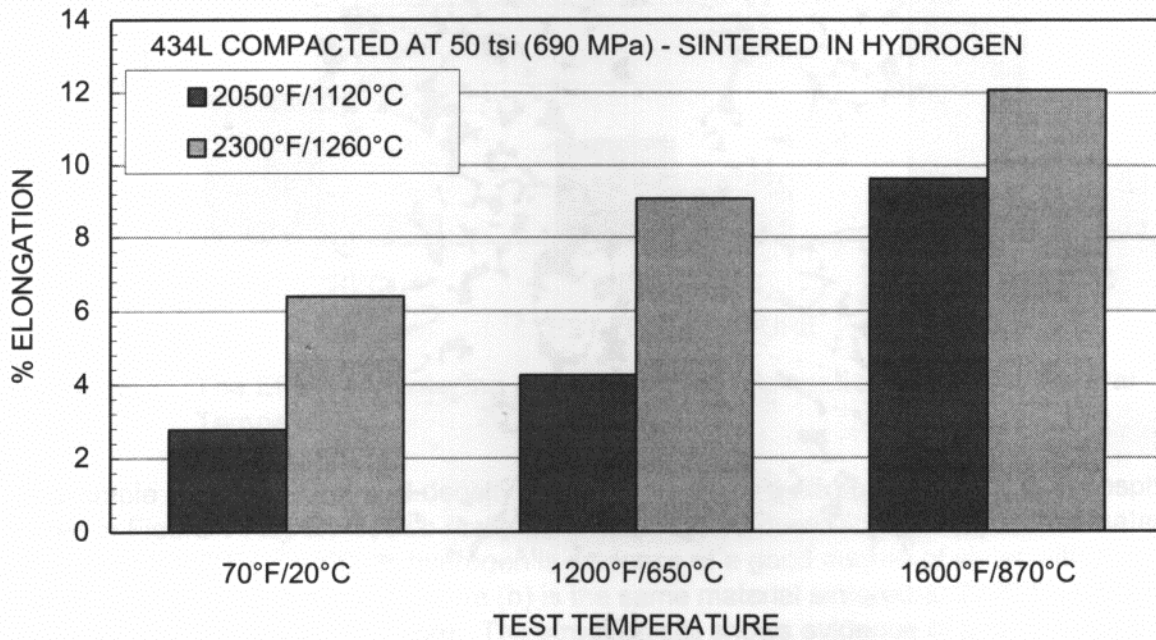


Figure 16: The Effect of Sintering Temperature on Elongation Tested at Several Temperatures

In order to assess the P/M grades as potential materials in automotive exhaust applications, a comparison was made for 409Cb, 434Cb and 434L with close equivalent wrought chemistry

counterparts. Published values for 409, 439 and 18Cr-Cb wrought grades were used and compared with P/M 409Cb, 434Cb and 434L, respectively, following compaction at 50 tsi (690 MPa) and sintering at 2300°F(1260°C) in hydrogen. [6]

The results for 0.2% offset yield strength (Figures 17,18 and 19) demonstrate that the P/M grades perform favorably to the wrought materials particularly at the higher test temperatures. The 409Cb and 434L P/M grades exceed the yield strength of the wrought counterparts at all of the test temperatures. The 434Cb lags behind the wrought 18Cr-Cb material at room temperature but performs equivalently at 1200°F (650°C) and exceeds the performance of the wrought material at 1600°F (870°C).

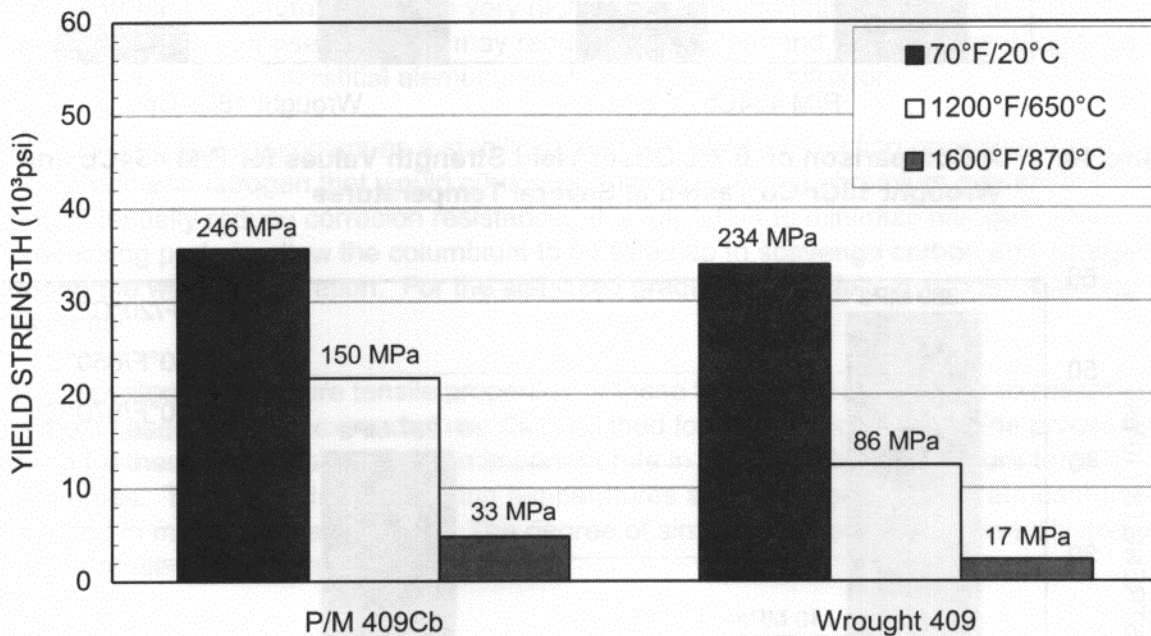


Figure 17: A Comparison of 0.2% Offset Yield Strength Values for P/M 409Cb and Wrought 409 Tested at Several Temperatures

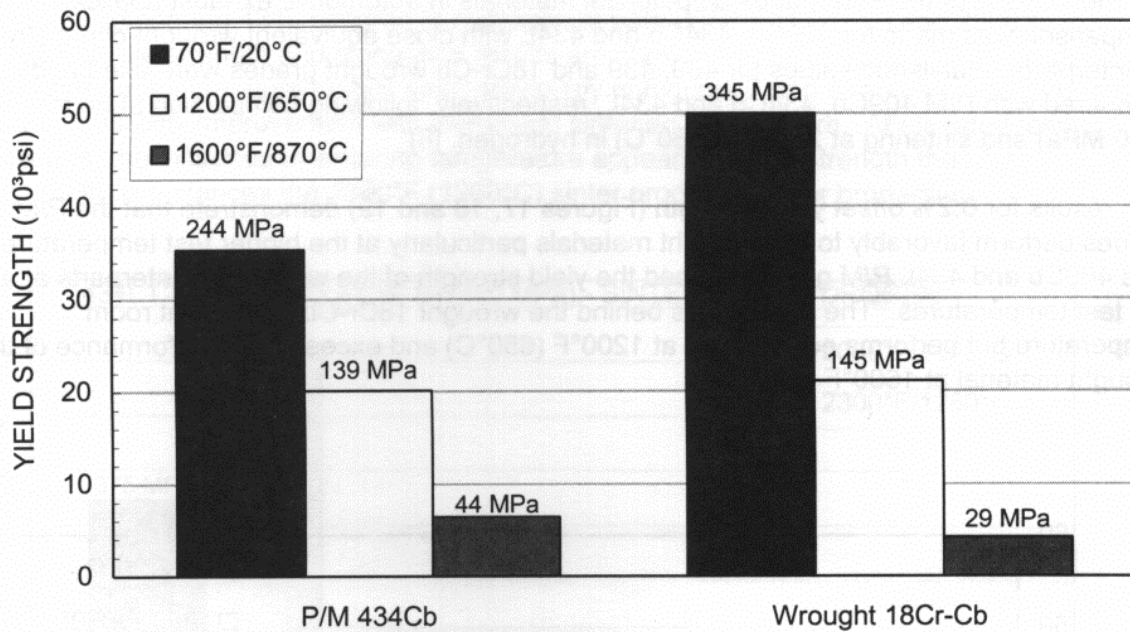


Figure 18: A Comparison of 0.2% Offset Yield Strength Values for P/M 434Cb and Wrought 18Cr-Cb Tested at Several Temperatures

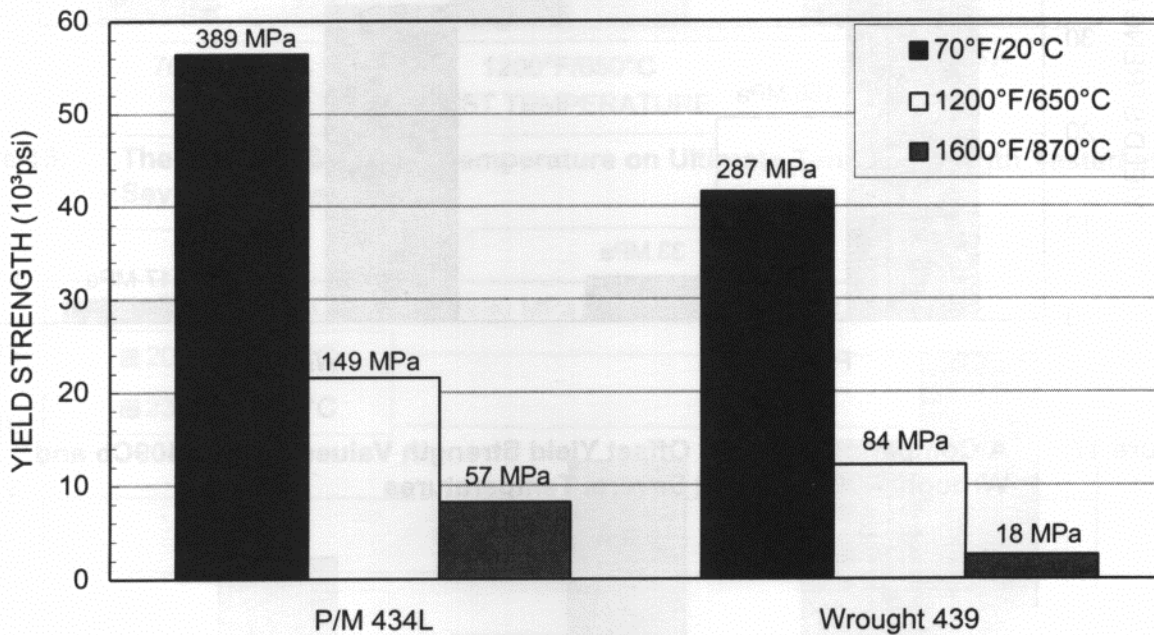


Figure 19: A Comparison of 0.2% Offset Yield Strength Values for P/M 434L and Wrought 439 Tested at Several Temperatures

CONCLUSIONS

This paper reviewed several important points in the processing and use of ferritic P/M stainless steels, including the following major items:

- Effect of base material and processing on basic mechanical properties.
- Optimal processing of columbium stabilized grades.
- Effect of service temperature on tensile properties and how the properties are affected by processing route.
- Comparison of P/M materials with wrought counterparts.

The following can be concluded from the study:

1. Elevated sintering temperatures result in both increased densification and increased mechanical properties. This is particularly the case with columbium stabilized grades where processing at lower sintering temperatures resulted in poor properties, with large improvements observed at higher sintering temperatures. Information presented here suggests that sintering temperatures of at least 2300°F(1260°C) are called for to optimize the properties and performance of these alloys.

2. For the materials in this study, the 434L material indicated the highest strength but the lowest ductility. When sintered at 2300°F(1260°C), the 410L and 409Cb materials both indicated good strength levels with very high elongation and impact properties. The presence of Cb in these materials may reduce the strength and increase the ductility as a result of tying up of interstitial elements such as carbon and nitrogen.

3. The presence of columbium as a stabilizing element during a welding operation helps to tie up carbon and nitrogen that would otherwise combine with the chromium present in the alloy and potentially reduce corrosion resistance. It is important to minimize nitrogen when processing parts to allow the columbium to be freed up to scavenge carbon and nitrogen during the welding operation. For the stabilized grades of materials, hydrogen sintering is essential.

4. The elevated temperature tensile properties of these ferritic P/M alloys were excellent and, in most cases, actually exceeded results published for wrought materials. The processing route for these grades also plays an important role in the elevated temperature tensile properties. The use of lower sintering temperatures and a nitrogen bearing atmosphere resulted in much reduced ductility. The degree of sinter was shown to be severely impacted by the presence of nitrogen in the material.

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