EFFECT OF PROCESSING PARAMETERS ON THE ROOM AND ELEVATED TEMPERATURE MECHANICAL PROPERTIES OF P/M 409L AND 434L STAINLESS STEELS

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ABSTRACT

P/M ferritic stainless steels 409L and 434L are most commonly specified for automotive exhaust system components. A sintered density of 7.25 –7.30 g/cm^3 is desired in these applications, in order to meet mechanical property, as well as, leak tightness, thermal fatigue, oxidation resistance and weldabilty criteria. With each of these two alloys, the desired sintered density of 7.25- 7.30 g/cm^3 is achievable via various combinations of compaction and sintering parameters.

This paper compares the room and elevated temperature mechanical properties, as well as the microstructure, of P/M 409L and 434L sintered to 7.25 – 7.30 g/cm^3 using various combinations of process parameters. Sintering temperatures employed range from 1232^{0}C (2250^{0}F) to 1366^{0}C (2490^{0}F).

INTRODUCTION

Currently, the single largest application for P/M 400 series stainless steels is in the manufacture of automotive exhaust flanges. Grades 409L and 434L are predominantly selected for this application. Since these flanges are structural components they are required to meet stringent mechanical property criteria. Additionally, these components are required to possess adequate resistance to corrosion, oxidation, and thermal fatigue. Hence, qualification of a P/M stainless steel in the flange application is not only based on mechanical properties, but also on satisfactory performance in OEM specified corrosion, oxidation, thermal fatigue and leak tightness tests [1, 2].

Work carried out by various P/M parts fabricators and powder producers in the mid 1990s have shown that the P/M stainless steel flanges need to possess a minimum sintered density of 7.20 g/cm^3 in order to meet the OEM specified performance criteria [3 - 5]. It has also been demonstrated that the material be sintered under conditions that result in low levels of residual nitrogen and carbon, thus making it imperative that the sintering atmosphere must be 100% hydrogen [6]. Hydrogen sintering is also desirable because it leads to a high degree of shrinkage, thus permitting the achievement of the desired sintered density utilizing commonly available sintering temperatures. For example, sintering temperatures as low as 1232^{0}C (225^{0}F) can be practically employed to achieve the desired sintered density without raising the compaction pressure to an unreasonably high level.

Sintering temperature can have a profound influence on the mechanical properties of a P/M stainless steel. Sintering temperature and time determine the degree of particle bonding, pore rounding, grain size, and to a lesser extent, the residual levels of oxygen and carbon - all of which influence the mechanical properties of the material. For example, Tunberg et al. [7] observed that when sintering temperature was increased from 1200^0C (2190^0F) to 1250^0C (2280^0F) in the vacuum sintering of 304L, tensile strength increased by 10%, ductility increased by 44% and impact strength increased by 30% (a small portion of these improvements is attributable to the increase in sintered density). Rawlings et al. [8] noted a 40% increase in tensile strength, and a four fold increase in impact energy for 409L when sintering temperature was increased from 11210^0C (2050^0F) to 1260 ^{0}C (2300 ^{0}F), for the same sintered density (6.90 g/cm^3).

Because these components are exposed to elevated temperatures in service, elevated temperature mechanical properties are taken into consideration in designing. The elevated temperature yield strength of a material is indicative of its stress relaxation behavior. This characteristic of the material determines how well a flange will be able to retain the clamping force of a flange-pipe assembly at elevated temperatures, over long periods of time; and as such it is a critical parameter for ensuring the leak tightness of the assembly.

Achievement of the desired sintered density of 7.25 – 7.30 g/cm^3 is possible with multiple combinations of compaction pressure and sintering temperature. The choices become wider as individual parts makers acquire larger presses, and newer furnaces with higher temperature capabilities. The actual combination of process parameters selected is most often based upon the overall process cost and operational conveniences.

While the minimum required sintered density has been determined as 7.20 g/cm^3, most parts makers prefer to keep the sintered density in the range of 7.25 to 7.30 g/cm^3 in order to make allowance for any part-to-part and within a part variation in density.

This paper examines the relative effects of the compaction pressure and sintering temperature on the mechanical properties of 409L and 434L for sintered densities in the range of 7.25 to 7.30 g/cm^3. Compaction pressures selected were 483, 552, 690 and 770 MPa (35, 40, 50 and 56 TSI), which led to specific sintering temperatures that fell in the range of 1227 ^{0}C to 1366 ^{0}C (2240 ^{0}F to 2490^0F).

MATERIALS

Commercially available, water atomized 409L and 434L powders were used in the study. Both were annealed powders with a nominal particle size of minus 100mesh. Their chemical compositions conform to the specifications set by MPIF Standard 35. Properties of these powders are listed in Table 1. Both powders were lubricated with AcrawaxC lubricant; the amount of lubricant added was 0.75%, by weight.

Table 1: Compositions and properties of powders used (wt %)

Grade	Cr	Mo	Cb	Mn	Si	P	S	C	O	N	Fe	A. D., g/cc	<325 mesh
409L	11.5	0	0.55	0.22	0.94	0.014	0.015	0.022	0.177	0.029	Bal.	2.78	36
434L	16.8	0.9	0	0.18	0.83	0.013	0.015	0.028	0.29	0.028	Bal.	2.8	40

PROCESSING

Since the test plan included elevated temperature tensile testing, 'dog bone' tensile test specimens were considered unsuitable for the study. Instead, the sintered samples processed were in the form of Izod Impact Test specimens, which could be conveniently machined into ASTM "Machined 190-Round Tension Specimens" (ASTM B 925-03). Compaction pressures selected were 483, 552, 690 and 772 MPa (35, 40, 50 and 56 TSI). Employing these four compaction pressures grade 409L and 434L powders were compacted into the shape of the standard Izod Impact Test specimens (10mm x 10mm x 75mm). Green densities achieved at various compaction pressures are listed in Table 2 and plotted in Fig. 1.

Green samples were delubricated at 538 ^{0}C (1000 ^{0}F) under a dissociated ammonia atmosphere, using a mesh belt furnace. The total time period for heat up and delubrication was 20 minutes. Sintering was carried out in a laboratory pusher furnace under an atmosphere of 100% hydrogen. Sintering time period was 30 minutes at temperature, with the total time period in the hot zone being around 45 minutes. Sintering temperature was adjusted for each sample set (i.e. each grade and green density combination) so that the resulting sintered densities fell in the range of 7.24 and 7.30 g/cm^3. Any sintered bar whose density did not fall in this range was not included in the test program. Table 2 lists the actual sintering temperatures employed and the resulting sintered densities, for each of the eight sample sets.

Table 2: Compaction and sintering parameters

Alloy grade	Compaction pressure MPa (TSI)	Green density, g/cm3	Sintering temperature,	Sintered density, g/cm^3	Sample set
409L	483 (35)	6.36	1366 ^{0}C (2490 ^{0}F)	7.24 –7.28	409L - A
409L	552 (40)	6.42	1321^{0}C (2410^{0}F)	7.25 – 7.30	409L – B
409L	690 (50)	6.60	1271^{0}C (2320^{0}F)	7.24 – 7.27	409L – C
409L	772 (56)	6.66	1238^{0}C (2260^{0}F)	7.25 – 7.27	409L - D
434L	483 (35)	6.20	1360^{0}C (2480^{0}F)	7.27 – 7.30	434L - E
434L	552 (40)	6.31	1316^{0}C (2400^{0}F)	7.29 – 7.30	434L - F
434L	690 (50)	6.54	1260 ^{0}C (2300^{0}F)	7.27 – 7.30	434L - G
434L	772 (56)	6.60	1227 ^{0}C (2240^{0}F)	7.27 – 7.30	434L - H

For each sample set, sufficient number of sample bars were sintered to permit tensile testing at the three selected temperatures, in duplicate, as well as for conducting impact testing, microstructural evaluation, and analysis of carbon, nitrogen and oxygen contents.

Although no emphasis was placed on the effect of compaction pressure on the green strengths of the two powders, for the sake of reference the green strengths, at the compaction pressure of 552 MPa (40 TSI), were 12.2 MPa (1770 psi) for 409L and 10 MPa (1440 psi) for 434L,

TESTING

The sintered Izod Impact Test specimens were machined into standard ASTM Machined 190-Round Tension Test specimens (4.75 mm dia. x 25.5 mm, 0.187 inch dia. x 1.00 inch, with threaded grip sections as described in ASTM Standard B 925-03). Tensile testing was carried out in accordance with ASTM test method E21-98. All machining and testing was carried out at Westmoreland Mechanical Testing and Research, Inc., Youngstown, Pa.

Based on the specifications set forth by one US automobile manufacturer, the test temperatures selected were: room temperature, 649^{0}C (1200^{0}F) and 871^{0}C (1600^{0}F). All sample sets were tested in duplicate, at each of the three test temperatures. All elevated temperature mechanical tests were carried out in an atmosphere of argon. Test specimens were soaked for 30 minutes at the test temperature prior to actual testing.

Charpy impact tests were carried out at room temperature utilizing un-notched specimens, in accordance with ASTM test method E23-02.

Samples from each sample set were analyzed for carbon content using LECO Model CS-244 Carbon and Sulfur Analyzer, and for oxygen and nitrogen contents using LECO Model TC-436 Oxygen and Nitrogen Analyzer.

RESULTS AND DISCUSSION

For each compaction pressure, it was noted that the green densities of 434L samples were slightly lower than those of 409L samples. This would be expected because of the higher alloy content of 434L. The differences in the green density were greater at the lower compaction pressures, as compared to the higher compaction pressures (Table 2 and Fig. 1).

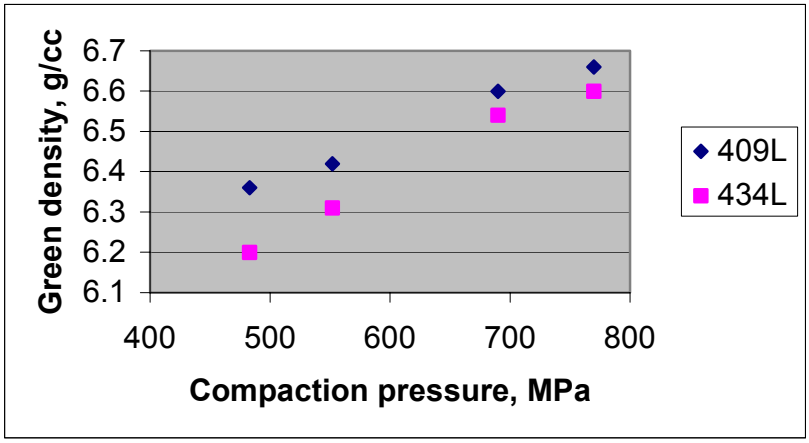


Fig. 1: Green density versus compaction pressure for 409L and 434L

For each of the four compaction pressures, the rate of densification during sintering, was found to be greater for 434L, as compared to 409L; thus requiring a slightly lower sintering temperature than what was necessary for 409L for attaining the desired sintered density. Despite these adjustments sintered densities of 434L samples were slightly higher than those of 409L, by about 0.02 g/cm^3.

Table 3 lists the results of the carbon, oxygen and nitrogen analyses for the eight sample sets. As may be seen here, the nitrogen contents of all sample sets were desirably low, as would be expected of hydrogen sintered P/M stainless steels.

Table 3: Results of the carbon, oxygen and nitrogen analyses

Sample set	Sintering temperature, ^{0}C (^{0}F)	Compaction pressure, MPa (TSI)	Carbon content, %	Oxygen content, ppm	Nitrogen content, ppm
409L - A	1366 (2490)	483 (35)	0.006	1674	281
409L - B	1321 (2410)	552 (40)	0.009	2222	355
409L - C	1271 ((2320)	690 (50)	0.008	2084	340
409L - D	1238 (2260)	772 (56)	0.042	3240	355
434L - E	1360 (2480)	483 (35)	0.005	1931	388
434L - F	1316 (2400)	552 (40)	0.011	2351	233
434L - G	1260 (2300)	690 (50)	0.014	2570	357
434L - H	1227 (2240)	772 (56)	0.052	3328	366

The oxygen and carbon contents of sample sets representing the three lower compaction pressures are at satisfactory levels. The carbon contents are well below the 0.03% limit of the L grade specification. Oxygen contents are below 3000 ppm, which is also satisfactory. The sample sets representing the lowest compaction pressure (highest sintering temperature) are found to have undergone significant reduction of oxygen and carbon during sintering. This is attributed to the greater kinetics of oxide reduction, by both carbon and hydrogen, occurring at the elevated temperature.

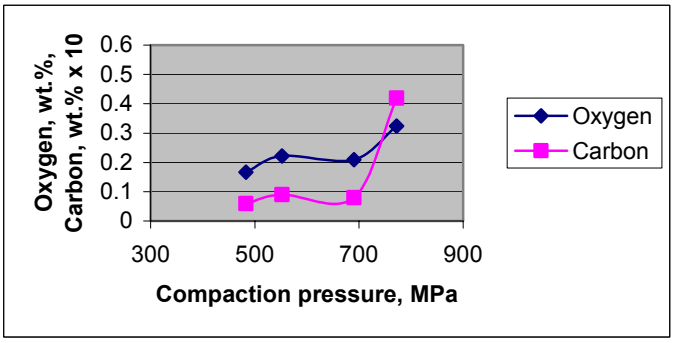


Fig. 2: Carbon and oxygen content of 409L samples as a function of compaction pressure.

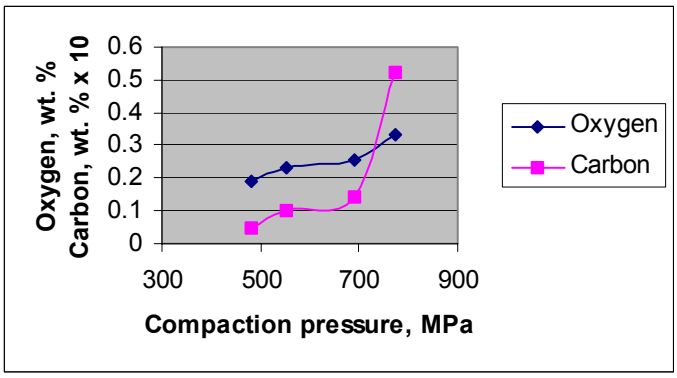


Fig. 3: Carbon and oxygen content of 434L samples as a function of compaction pressure.

The carbon and oxygen contents of the sample sets representing the highest compaction pressure are higher than desirable, especially with regard to the carbon content (Fig. 2 and 3). In both cases carbon contents are greater than the 0.03% specification set for L grade stainless steels. These high residual

carbon contents are attributed to inadequate delubrication of the green samples, resulting most likely from their high green densities. Since all samples were delubricated under the same furnace conditions (which represented typical commercial practice), it is clear that the use of such high compaction pressures necessitates extra ordinary efforts for adequate delubrication. The high residual oxygen content is attributed to both incomplete delubrication,, and slower kinetics of the oxide reduction at the lower sintering temperature.

Table 4 lists the results of the mechanical testing of 409L samples at various test temperatures. The room temperature tensile and yield strengths are found to be unaffected by a change in sintering temperature. Ductility and impact energy are lower for the sample set representing the highest compaction pressure (lowest sintering temperature). At the lowest sintering temperature (1238^0C, 2260^0F), the tensile elongation is 9%, which is considered satisfactory. A minimum elongation of 7% is usually specified for the exhaust flange application. Similarly, the impact energy of 105 Joules is considered satisfactory for the application.

Table 4: Results of tensile testing of 409L sample sets

Sample set	Compaction pressure MPa(TSI)	Sintering temperature ^{0}C (^{0}F)	Test temperature, ^{0}C, (^{0}F)	UTS, MPa, (KSI)	YS, MPa, (KSI)	Elong. %	Impact energy Joules
409L- A	483(35)	1366 (2490)	RT	373 (54.1)	214 (31)	17	163
			649 (1200)	210 (30.4)	102 (14.8)	15	
			871 (1600)	32.4 (4.7)	23 (3.3)	62	
409L –B	552(40)	1321 (2410)	RT	366 “(53.1)	212 (30.7)	16	147+
			649 (1200)	253 (36.7)	114 (16.5)	12.5	
			871 (1600)	31.0 (4.5)	20.7 (3)	63	
409L –C	690(50)	1271 (2320)	RT	357 (51.8)	209 (30.3)	17	135
			649 (1200)	176 (25.5)	100 (14.5)	11	
			871 (1600)	29.0 (4.2)	18.6 (2.7)	55	
409L-D	772(56)	1238 (2260)	RT	377 (54.7)	208 (30.2)	9	105
			649 (1200)	155 (22.5)	103 (15)	10	
			871 (1600)	29.0 (4.2)	20 (2.9)	30	

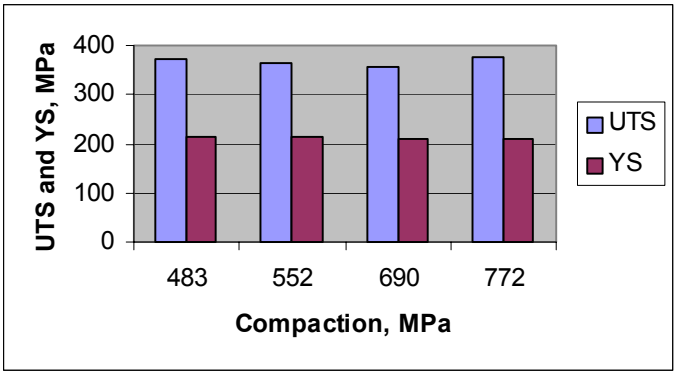


Fig. 4: Room temperature tensile and yield strengths of 409L samples as a function of compaction pressure.

As may be seen in Fig. 4, despite the higher residual carbon content of the Sample Set 409L – D, all sample sets exhibit very similar tensile and yields strengths. This is not the case for 434L, as will be discussed later. The relative insensitivity of 409L to greater than 0.03 % carbon content is evidentaly

due to the presence of columbium in the material. Columbium ties up carbon and nitrogen, thus preventing these interstitials from going into solution in the matrix or forming compounds with chromium. However, the total amount of carbon and nitrogen that can combine with columbium is one-eighth the amount of columbium present. In these 409L samples the columbium content was 0.55%, which would be effective as a stabilizer for up to 0.07% total carbon and nitrogen content. Sample Set 409L – D has a combined carbon and nitrogen content of around 0.077 %, and hence most of the carbon and nitrogen present in this sample set would have combined with the available columbium, thus eliminating the negative effects from their presence in the matrix.

Table 5 lists the results of mechanical testing of the 434L samples at various test temperatures. Room temperature ductility of the Sample Set 434L – H is 3%, which is significantly lower than the minimum ductility required for the application. Similarly, its impact energy of 42 Joules is also much lower than the desired value of 80 Joules. The ductility of the Sample Set 434L - G is 7%, which is considered borderline for the application.

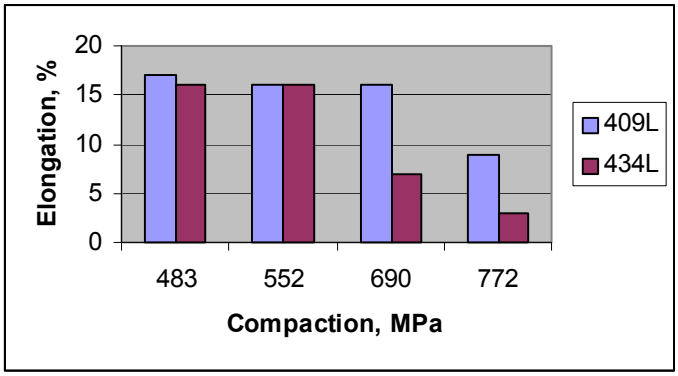


Fig. 5: Room temperature tensile elongations of 409L and 434L samples as a function of compaction pressure.

Table 5:Results of tensile testing of 434L samples

Sample Set	Compaction MPa (TSI)	Sintering Temp, ^{0}C (^{0}F)	Test Temperature, ^{0}C, (^{0}F)	UTS, MPa, (KSI)	YS, MPa, (KSI)	Elong. %	IE, J
434L- E	483(35)	1360 (2480)	RT	402 (58.3)	246 (35.6)	16	138
			649 (1200)	175 (25.4)	121 (17.5)	29	
			871 (1600)	35.8 (5.2)	23.5 (3.4)	66	
434L – F	552(40)	1316 (2400)	RT	405 (58.7)	248 (36.0)	16	147.+
			649 (1200)	169 (24.5)	118 (17.1)	29	
			871 (1600)	36.5 (5.3)	20.7 (3)	74	
434L – G	690(50)	1260 (2300)	RT	477 (69.2)	277 (40.2)	7	129
			649 (1200)	179 (25.9)	129 (18.7)	27	
			871 (1600)	36 (5.2)	23.4 (3.4)	52	
434L- H	772(56)	1227 (2240)	RT	512 (74.3)	329 (47.7)	3	42
			649 (1200)	207 (30.0)	143 (20.0)	20	
			871 (1600)	40 (5.8)	24 (3.4)	38	

The room temperature tensile and yield strengths are higher for sample sets 434L – G and 434L – H, as compared to those of the sample sets 434L – E and 434L – F (Fig. 6). In the case of Sample set 434L – H, the increase and strength and the decrease in ductility are attributable, in large, part to the high residual carbon content of 0.052%. In the absence of a stabilizer (such as columbium), this amount of residual carbon can lead to solution strengthening, and also martensite formation.

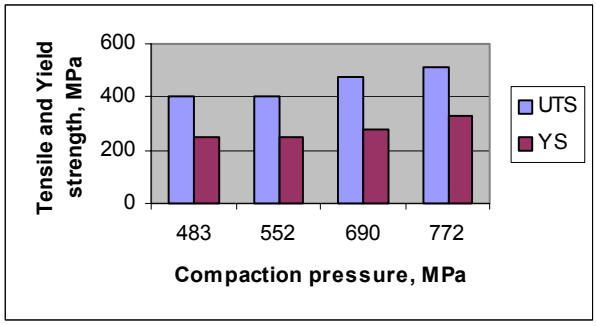


Fig. 6: Tensile and yield strengths of 434L sample sets as a function of compaction pressure.

Presence of martensite in the Sample set 434L – H was confirmed in metallographic examination. Fig 7 (a) thru 7(f) shows the microstructures of Sample Sets 409L – A, 409L – C, 409L – D, 434L – E, 434L – G and 434L – H. (Microstructures of 409L – B and 434L – G were similar to those of the 409L – A and 434L – E, respectively, and hence are not included.) Overall, the microstructures of 409L samples show the presence of many fine precipitates in the grain boundaries and within the grains. These precipitates are assumed to be compounds of columbium (carbides, nitrides). In contrast to these, the microstructures of 434L samples are largely free of such precipitates. Owing to their higher sintering temperature, the grain sizes of 409L – A and 434L – E are significantly larger than those of their lower sintering temperature counter parts. Some differences in the grain size are also noticeable between the samples representing the two lower sintering temperatures, for both alloys. Only the sample of 434L- H shows presence of the martensite phase, whose amount is estimated to be around 7%, by volume.

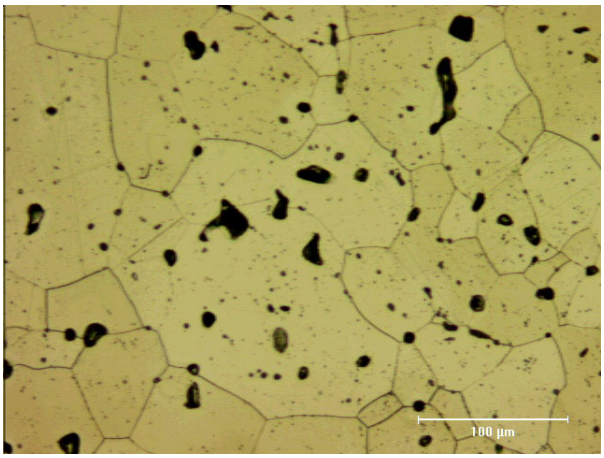


Fig. 7 (a): Photomicrograph of Sample Set 409L – A, showing large grains and fine precipitates in the grain boundaries and within the grains (etched).

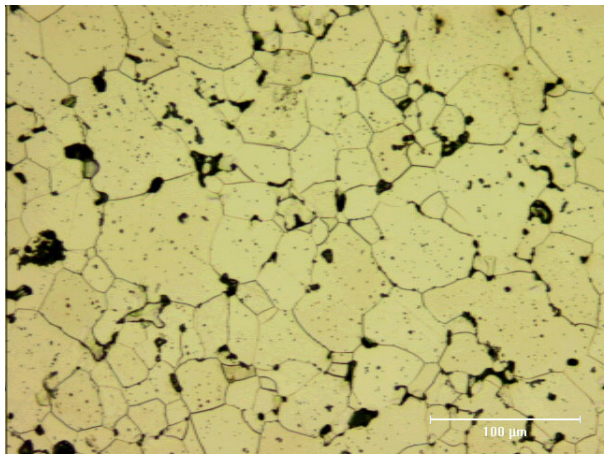


Fig. 7 (b): Photomicrograph of Sample Set 409L – C, showing small grains and fine precipitates in the grain boundaries and within the grains (etched).

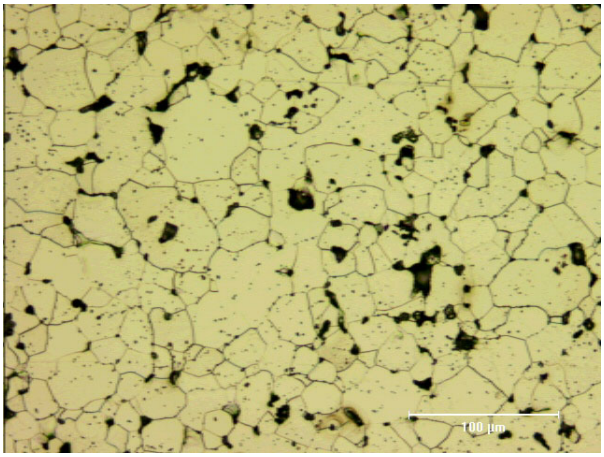
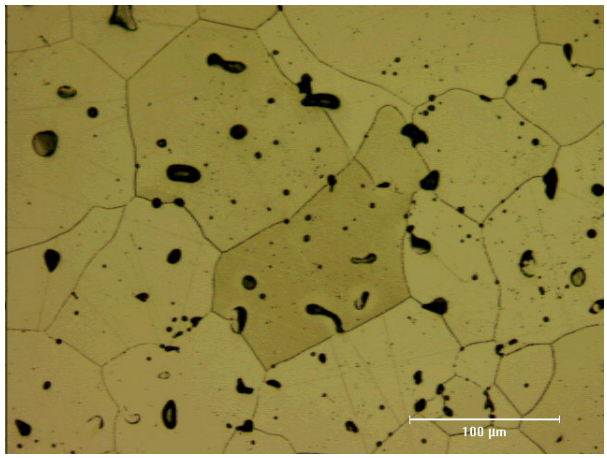


Fig. 7 (c): Photomicrograph of Sample Set 409L – D, showing small grains, angular porosity and fine precipitates in the grain boundaries and within the grains (etched).

Fig. 7(d): Photomicrograph of Sample Set 434L – E, showing large grains (etched).



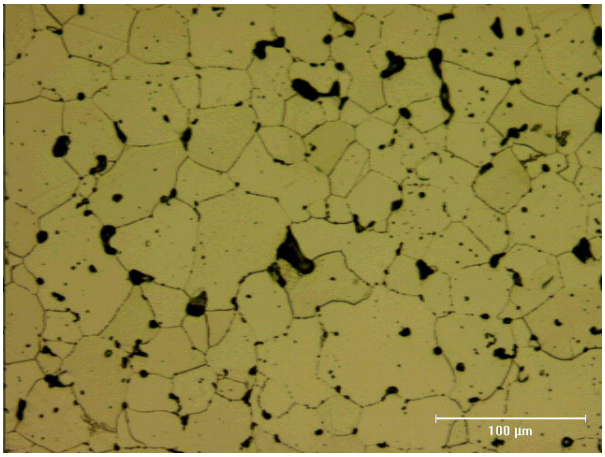


Fig. 7(e): Photomicrograph of Sample Set 434L – G, showing small grains (etched).

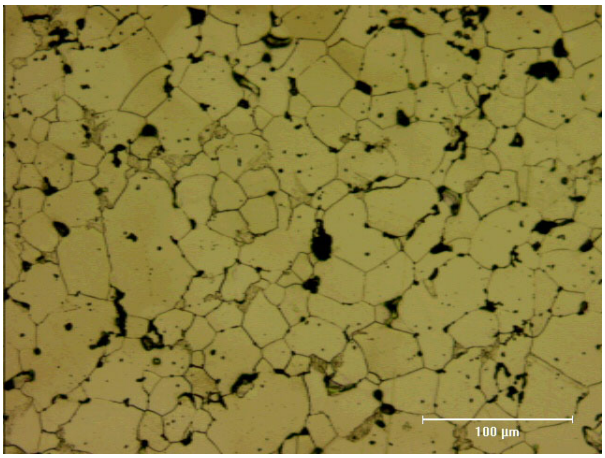


Fig. 7(f): Photomicrograph of Sample Set 434L – H, showing presence of martensite, angular porosity and small grain size (etched).

The increase in strength of 434L – G over that of 434L –E and 434L – F is attributed to its finer grain size.

In the absence of a stabilizer, high residual carbon contents are also undesirable from the corrosion resistance point of view.

A comparison of the microstructures of the samples indicates that as the degree of pore rounding is reduced as the sintering temperature is decreased. Angular porosity is clearly visible in the microstructures of sample sets 409L – D and 434L – H. Lack of pore rounding is considered to have contributed to the lower ductility and impact energy of these two sample sets.

The elevated temperature tensile and yield strengths of 409L samples are practically unaffected by the process parameters. These elevated temperature yield strengths are very similar to those reported by Lee et al. and Hubbard et al. [2, 5].

The elevated temperature tensile and yield strengths of 434L samples are somewhat higher for the lower temperature sintered sample sets, namely 434L – G and 434L – H, when compared to the other two sample sets. The enhancements in the room temperature mechanical strengths of these two samples are partially retained at the elevated temperatures.

The 400 series P/M stainless steels exhibit higher ductility at elevated temperatures, and as result, all sample sets are found to possess satisfactory ductility at higher test temperatures. The significantly low room temperature ductilities of Sample Sets 434L – G and 434L – H have been largely overshadowed by the favorable effect from elevated temperature testing.

The most significant observation of this study is the reduced room temperature ductility of 434L samples processed by employing the highest compaction pressure and the lowest sintering temperature. Ductility is essential in this application because of two reasons: most exhaust flanges are expected to undergo some bending during assembly; and secondly ductility has a significant influence on the material's resistance to thermal fatigue. Since these components undergo thermal cycling in service, resistance to thermal fatigue is considered a highly essential requirement. Loss of ductility is primarily the result of higher level of residual carbon. However, it is also evident that angular pore structure and a small grain size have significantly contributed to the reduction in ductility. Both of these factors are the result of a lower sintering temperature. Sample Set 434L - G, which had sufficiently low residual carbon content had moderately low ductility, and this is attributed to tits small grain size and angular porosity.

Alloy 409L, by virtue of its columbium content, is found to be relatively unaffected by the process parameters, as long as the sintering temperature is kept above 1238^0C (2260^0F), and the sintered density is kept at or above 7.25 g.cm^3.

CONCLUSIONS

Although a sintered density of 7.25 g/cm^3 can be achieved via various combinations of compaction and sintering parameters, the use of compaction pressures above 690 MPa (50 TSI) can lead to significant reduction in ductility and impact energy in P/M 434L. The loss in ductility and impact energy primarily stem from inadequate delubrication, which leads to high residual carbon contents in the material; and to a lesser extent from the angular pore structure and small grain size that result from a relatively low sintering temperature.

P/M 409L is relatively unaffected by the process parameters in the range utilized in this study, primarily owing to the presence of columbium in the material. The tensile and yield strengths of P/ M 409L, as well as its ductility and impact energy are found to be satisfactory for all combination of process parameters studied.

The elevated temperature mechanical properties are relatively unaffected by the process parameters for both 409L and 434L.

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