EFFECTS OF CHEMISTRY VARIATIONS ON DIMENSIONAL CONTROL OF 316L STAINLESS STEEL

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ABSTRACT:

This research aims at understanding the relationship between powder chemistry, oxide species, sintering atmosphere and sintering response in compaction grade 316L stainless steel.This will lead to development of a process control tool to obtain higher dimensional precision andbroaden the market for P/M stainless steels. Six different lots of 316L stainless steel were compacted, sintered, and measured for dimensional change. Correlation values were drawn between the dimensional change and bulk chemistry, surface chemistry, and particle size. The data indicate a strong influence of surface chromium content, bulk manganese content, bulk carbon content, and D_{10} particle size on shrinkage.

INTRODUCTION:

Stainless steels have been processed by P/M for the past 55 years. The 300 series alloys are used extensively for applications where good corrosion resistance is required, while the 400 series alloys are used where magnetic properties, thermal conductivity, or thermal cycling are required [1]. Dimensional variation in sintering of stainless steels is a result of a combination of temperature, time, chemistry, oxygen species, and particle size variations. Larsen and Thorsen [2] have studied gas-metal reactions during sintering of 316L. SiO_2 was found to be the most stable oxide in 316L. Cr_2O_3 and $MnCr_2O_4$ are more easily reduced. Carbon was found to be a more effective reducing agent at high temperatures than dry hydrogen. Without carbon addition, 0.14wt% oxygen remained after sintering at 1250°C for 2 h in hydrogen, indicating that not all oxides were removed by hydrogen at the sintering temperature. With 0.1wt% carbon addition, the sintered oxygen content dropped to 0.03wt%. McMahon and Reen [3] modeled sintering

shrinkage of 316L powders vs chemistry using regression analysis. Using the model, they were able to predict dimensional change of powder lots not included in the regression within 95% tolerance limits.

EXPERIMENTAL PROCEDURE:

Six different lots of –100 mesh water atomized 316L stainless steel were obtained from several vendors. Each lot was measured for particle size using a Horiba laser scattering particle size analyzer. Bulk chemistry of the powders was measured by ICP analysis, while powder surface chemistry was measured by X-ray photospectroscopy. Carbon and oxygen contents were measured by combustion. Powders were mixed with 1.0 wt% Acrawax C in a Turbula blender for 20 minutes. Compaction was performed in a Gasbarre 60 ton hydraulic press. Compaction pressure was adjusted for each lot to yield a green density of 6.4 g/cm^3+/-0.03. Each sample was measured for height and diameter using a micrometer, and weighed on a laboratory balance. Debinding was performed in a retort furnace by heating in hydrogen at 10°C/min to 200°C, then 5°C/min to 475°C for 1 h [4]. Dilatometry of selected lots was performed in an Anter vertical dilatometer at 1150°C and 1300°C for 1 h in hydrogen. Sintering was performed in a CM Furnaces box furnace by heating at 10°C/min in hydrogen to 1150°C or 1300°C for 1 h. Thirty parts from each lot were sintered in each run. Parts were measured at each stage of processing for diameter and length with a micrometer, off burr. The correlation between the individual variables and the average shrinkage was calculated. The correlation coefficient, r, is a measure of linear association between two variables. It has a range of -1 to +1; positive values indicate a positive correlation (positive slope) and negative values indicate a negative correlation (negative slope). Values closer to ±1 indicate a stronger correlation.

RESULTS AND DISCUSSION:

The particle size distributions from each lot are given in Table I. The bulk chemical analysis and surface chemical analysis are given in Table II.

Table I.
Particle Size Analysis

Grade	D_{10}	D_{50}	D_{90}
1	16.6	41.2	81.7
2	15.1	41.5	91.5
3	17.4	44.3	77.6
4	18.6	41.7	81.7
5	18.1	42.9	85.8
6	13.5	34.9	78.0

Table II
Bulk and Surface Chemistry

Grade	Cr	Mn	Mo	Ni	Si	C	O	Cr*	Ni*	Si*	Fe*
1	16.6	0.16	2.36	13.9	0.86	0.024	0.22	1.3	0.1	15	3.0
2	16.7	0.16	2.16	13.4	0.90	0.021	0.21	1.6	0.1	15	2.9
3	16.2	0.19	2.08	12.1	0.80	0.024	0.22	1.8	0.5	17	2.9
4	16.2	0.17	2.14	12.2	0.73	0.019	0.27	2.1	0.1	17	2.9
5	16.2	0.12	2.24	13.3	0.62	0.016	0.29	2.2	0.1	15	3.3
6	16.1	0.12	2.15	13.1	0.56	0.010	0.26	3.2	0.1	22	1.7

*surface chemistry

Dilatometry results of selected grades all displayed similar shrinkage behavior. Figures 1 and 2 are dilatometry curves from grade 1 heated to 1150°C and 1300°C, respectively. In both cases, significant shrinkage occurs during the one hour hold time, indicating the importance of time at temperature in dimensional control. Higher temperature imparted higher shrinkage, as expected.

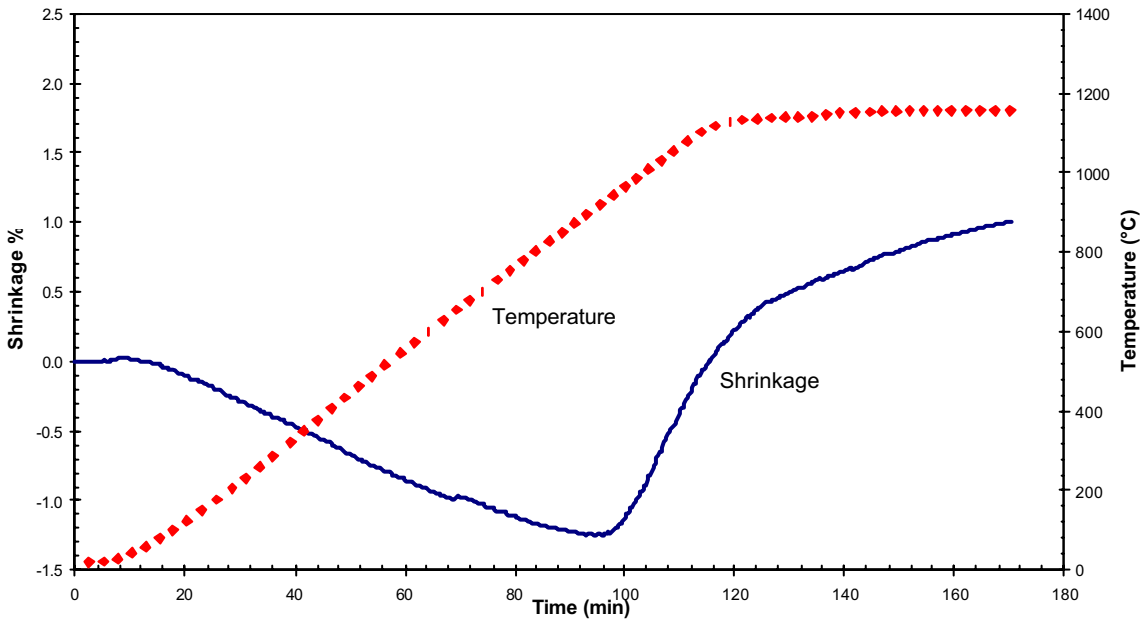

Figure 1. Dilatometry of 316L grade 1. Sample heated at 10°C/min to 1150°C for 1 h in hydrogen.

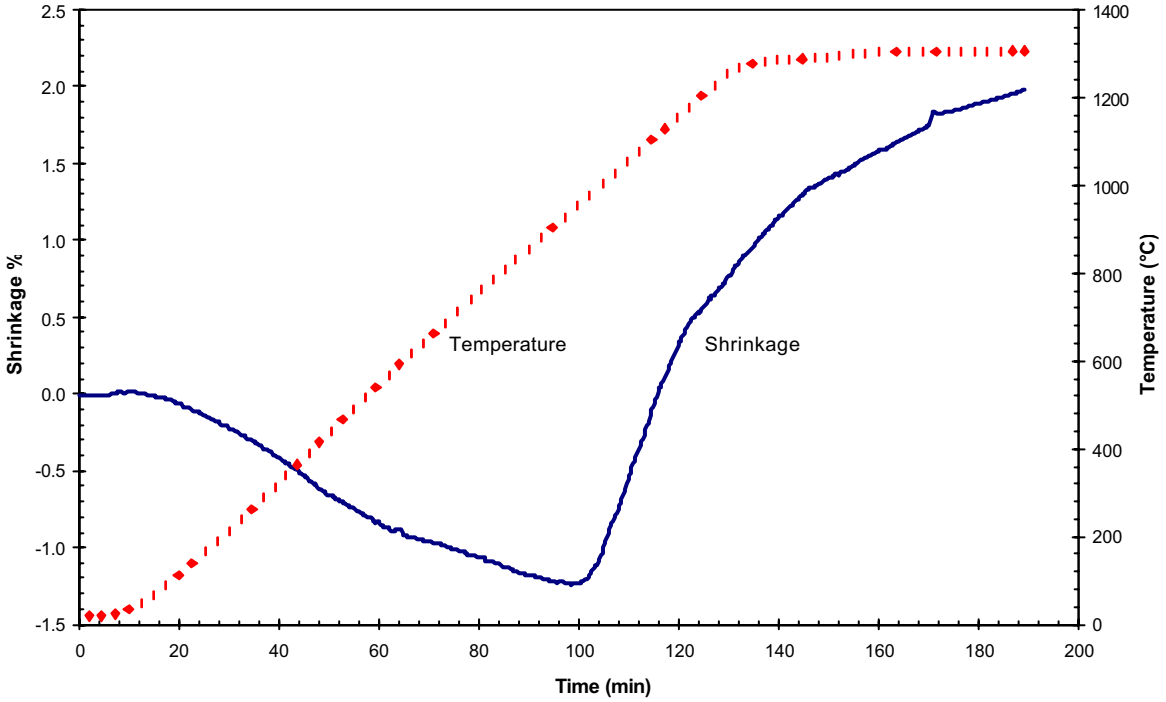

Figure 2. Dilatometry of 316L grade 1. Sample heated at 10°C/min to 1300°C for 1 h in hydrogen.

Sintering of 30 samples from each lot in a box furnace resulted in a dimensional precision of 0.04 standard deviation on the 12.7 mm cylinder diameter. The average shrinkage from each lot was plotted against each chemical element and combinations of chemical elements and evaluated for linear correlation. A -0.95 correlation was observed with Mn content at 1150°C, shown in Figure 3. A +0.90 correlation was observed at 1300°C with surface Cr content, shown in Figure 4, but not bulk Cr content. A non-linear increasing shrinkage was observed with decreasing D_{10} particle size at 1300°C, as observed in Figure 5. Figure 6 displays a -0.92 correlation between shrinkage and bulk carbon content. The anisotropic nature of the shrinkage arises from the fact that irregular shaped particles will tend to lie flat when poured into a die. This increases the contacts in the pressing direction, thus increasing shrinkage in that direction [5].

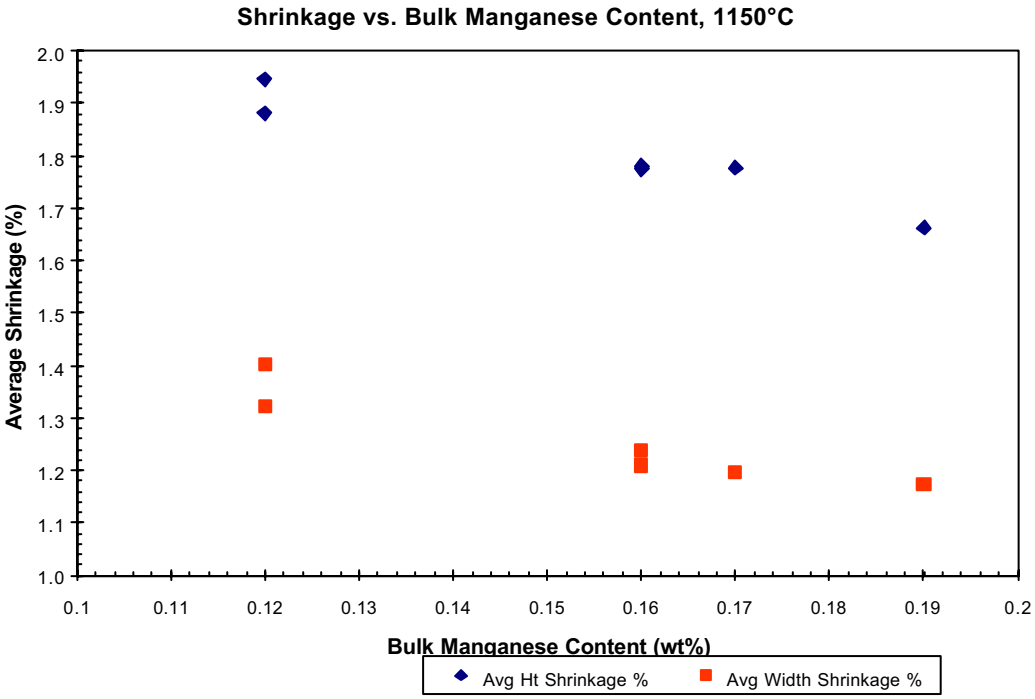

Figure 3. Shrinkage vs. bulk Mn content at 1150°C.

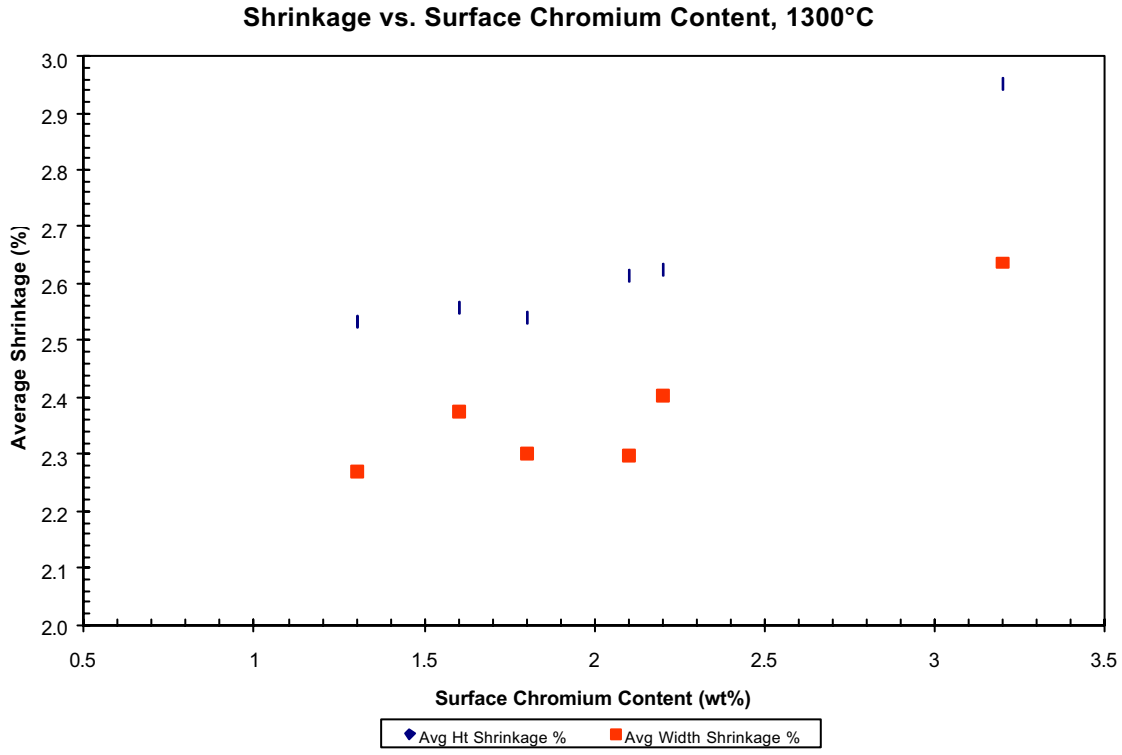

Figure 4. Shrinkage vs surface Cr content at 1300°C

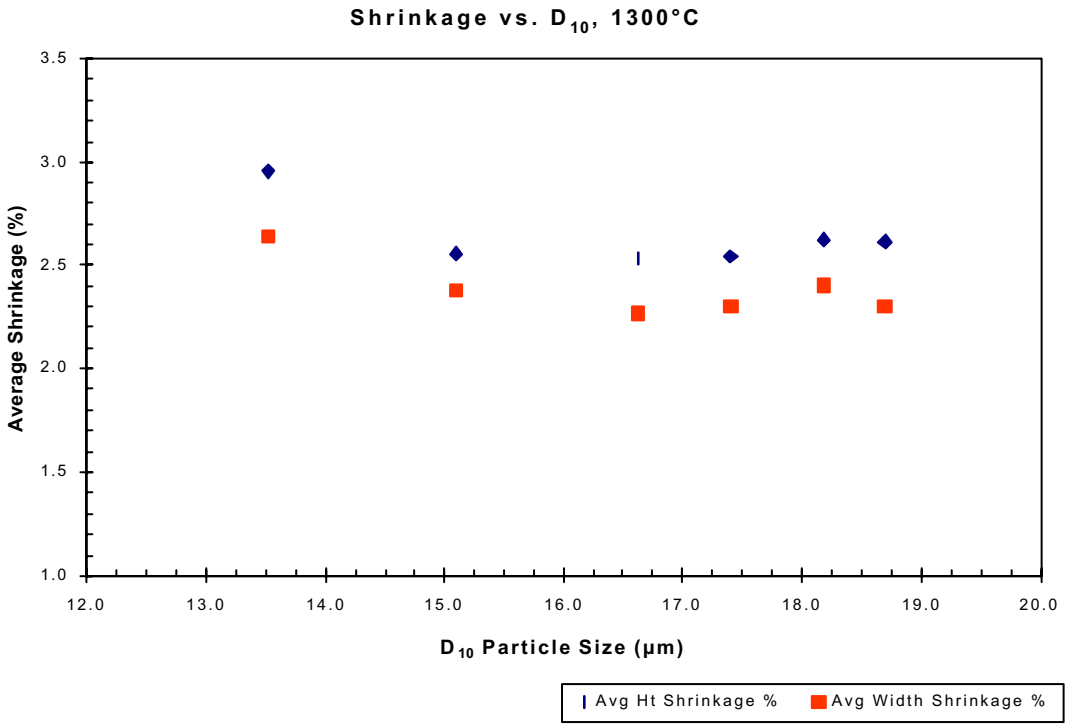

Figure 5. Shrinkage vs. D_{10} particle size at 1300°C.

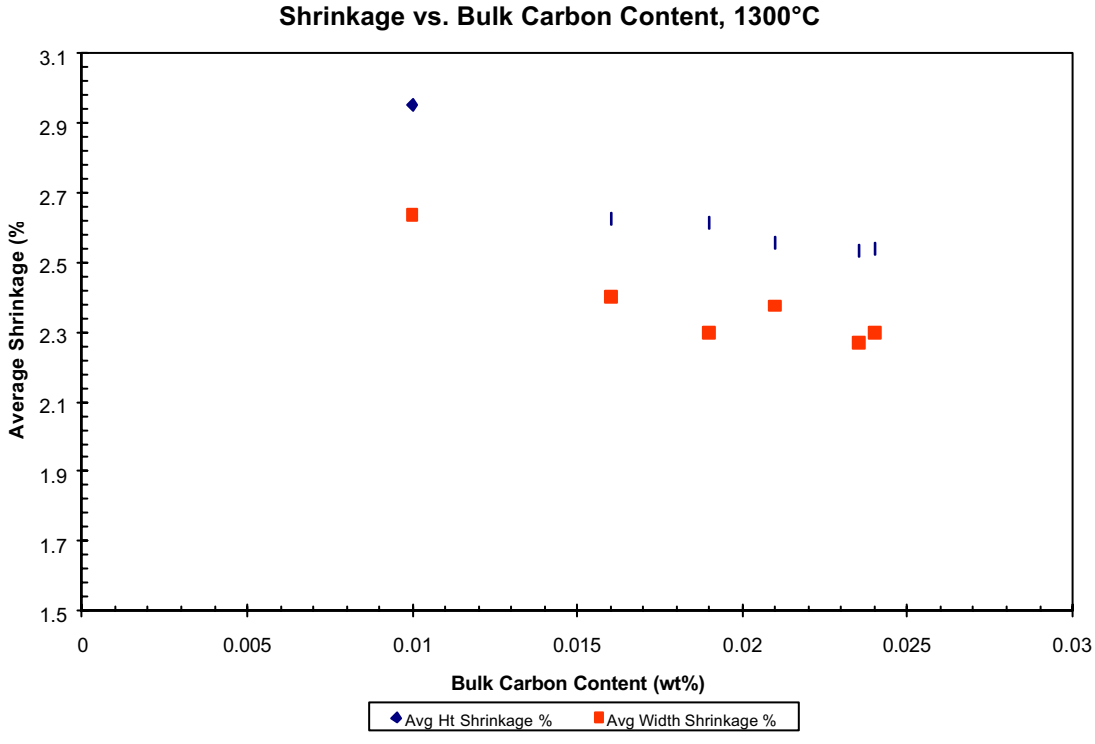

Figure 6. Shrinkage vs. bulk carbon content at 1300°C

CONCLUSIONS:

This research demonstrates the influence of variations in bulk chemistry, surface chemistry, and particle size on the shrinkage of 316L –100 mesh compacted powder. Results indicate that D_{10} particle size, Mn, surface Cr, and carbon all appear to have significant effects on shrinkage. Because each variable was not varied independent of the others, strong correlations may have been masked. It is not believed that firm conclusions can be drawn at this stage of the research.

FUTURE WORK:

Improved powder sampling is required for better isolation of variables. One lot will be sieved and re-blended to create different particle size distributions with one chemistry. Carbon to oxygen ratio will also be varied within selected lots. In addition to processing of new lots to increase the number of data points, stepwise regression analysis will be used to identify the effects of each variable. In this system with multiple factors affecting a single factor (shrinkage), it is necessary to determine which of the factors have the greatest influence on the response. Stepwise regression and best subsets (maximum R-squared) tests will be performed on the data. These are standard statistical methods for determining which factors influence a response variable in multiple linear regression. Other relationships in addition to pure linear relationships can be evaluated with these tests (logarithmic, power, hyperbolic, exponential, etc), since the parameters for the tests are still linear even if the factor variables are not.

ACKNOWLEDGEMENTS:

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