FERRITIC STAINLESS STEEL POWDERS FOR HIGH DENSITY APPLICATIONS

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Abstract: Most new applications for sintered stainless steels depend on high sintered density. To achieve this with close tolerances, the manufacturing process depends on optimised powder properties and high green density. This means that the ability to fill the die uniformly and at the same time keep the compressibility high are important. In this experimental approach different lubrication systems have been evaluated to get high sintered density with good tolerances.

Introduction

When producing high-density P/M stainless steel components the quality of the finished product depends strongly on the powder properties, which in turn affect properties such as weight scatter, density distribution and dimensional stability. These issues are especially important when high shrinkage during sintering cannot be avoided. Because of this procedure there is a risk of parts distortion due to uneven density distribution. To minimise this risk it is necessary to have a powder mix with very good filling capability. Of importance are both the tendency of segregation and the flow rate, which both are influenced by the base powder and the lubricant used.

In this paper an investigation on 409 Nb is described where most production issues regarding the powder mix are taken into account, such as flow rate, filling capability, compressibility, lubricant burn off, dimensional change during sintering.

Materials

All mixes included in the test plan are based on 409 Nb – 150 µm water atomised (not annealed) powder from Höganäs Belgium. The composition and physical properties can be found in table I.

Fe (%)	Cr (%)	Nb (%)	Si (%)	Mn (%)	C (%)	A.D. (g/cm³)	Hall Flow (s/50g)	-45 µm (%)	45 – 106 µm (%)	+106 µm (%)
Bal.	11.4	0.5	0.9	0.1	<0.01	2.68	29.5	40	48	12

Table I. Chemical composition and physical properties of the base powder (unlubricated).

Three lubricants were used; Kenolube™, Acrawax® C powder and Lithium stearate. Acrawax® C powder is an ethylene-bis-stearamide (EBS) lubricant, here after called Amide wax. Kenolube™ is a lubricant supplied by Höganäs AB, Sweden. The lubricants‘ particle size distribution was measured with laser diffraction, see Table II. The table shows statistical fraction values for 10, 50, 90 and 95 %.

Lubricant	x10	X50	x90	x95
Kenolube^{TM}	2.31	21.57	58.29	69.12
Li-stearate	1.15	4.92	20.50	28.92
Amide wax	6.07	29.2	55.24	64.55

Table II. Particle size distribution of the lubricants.

Lithium stearate has the finest particle size, which influences the powder properties to a large extent, especially the filling behaviour in production environments. Kenolube^{TM} and Amide wax have much larger average diameter, in the range of 20-30 µm. The high end of the distributions for Amide wax and Kenolube^{TM} are similar.

Six mixes, all with a total lubricant content of 1 %, were prepared according to table III.

Mix #	Base powder	Kenolube^{TM}	Lithium stearate	Amide wax
1	409 Nb	1.0 %	-	-
2	409 Nb	0.9 %	0.1 %	-
3	409 Nb	0.6 %	0.4 %	-
4	409 Nb	-	1.0 %	-
5	409 Nb	-	-	1.0 %
6	409 Nb	-	0.25 %	0.75 %

Table III. Mixes used in the investigation.

Experimental

The mixes were characterised with respect to apparent density and Hall flow. Furthermore, the compressibility, dimensional change and mechanical properties were determined using standard methods.

The filling characteristics for the investigated mixes were evaluated in a die-filling simulator (see figure 1), which simulates filling of die cavities of different sizes. The equipment includes 8 cavities with rectangular cross section having widths varying between 20 mm and 1 mm. Length along the filling direction and depth are 30 mm for all eight cavities. The filling shoe is moved from the left to the right in figure 1 followed by a return movement. With this sequence the die filling in real components, which often contain both wide and narrow sections, is simulated. The test equipment has been described before by Larsson and Vidarsson[1].

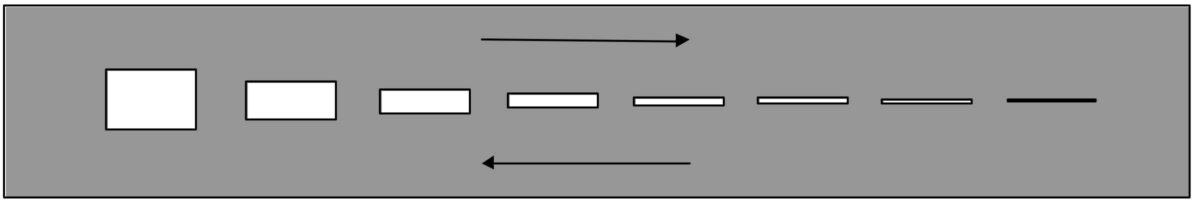


Figure 1. Sketch showing the 8 different cavities in the die-filling simulator.

In this investigation the speed of the filling shoe was varied. By weighing the powder in each cavity the fill density can be calculated and plotted versus the width of the cavities. The filling density is normally higher for a cavity with a larger width compared with a smaller one. A material with good filling characteristics exhibits small difference in filling density plotted versus the cavity width. To facilitate comparison between different mixes the filling is evaluated by calculating a filling index defined as the difference in filling density between the second largest (13 mm) and the third smallest (2 mm) cavity.

$$\text{Filling index} = 100 \text{ x } (AD_{13\text{ mm}}-AD_{2\text{ mm}})/\ AD_{13\text{ mm}}$$

A filling index of 0 % would be the ideal, but that is unattainable with real powder mixes. The filling index represents the quality of the filling in a component with openings in the cavity of different widths, which is often the case in real production. To get an even density distribution in production the filling index should be as low as possible. It should also be stable with regard to filling shoe speed. At high production rate the filling speed must also be high and this can result in uneven filling. A mix that fills evenly at low speed can behave poorly when the production rate is high. This factor can also be evaluated by studying the filling index diagram. When the filling shoe rate is increased above a certain value all mixes will eventually have worse filling characteristics.

The lubricant burn off characteristics were analysed by Thermo Gravimetric Analysis (TGA). In this case the equipment measured the weight change of small (0,5 g) powder mix samples during heating. The heating rate was 10°C/min from room temperature to 700°C. The atmosphere during the test was 96 % N_2/ 4 % H_2.

Tensile and impact energy test bars were compacted with 500, 650 and 800 MPa and the ejection force was measured during the ejection phase. The ejection force was integrated numerically to get a value on the total ejection energy needed for the different mixes. TRS bars were compacted at the same compaction pressures for the purpose of green strength testing by three-point bending. Sintering was carried out in a lifting hearth furnace at 1350°C for 30 minutes in 100 % hydrogen. The furnace was set to heat the material with 2°C per minute, which is quite slow compared to industrial practice, and as a result of this the shrinkages and sintered densities were very high.

Results

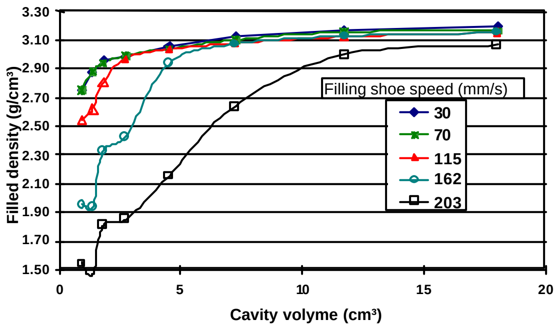
Table III shows the powder properties determined by Hall Flow and apparent density (AD) measurements. It also shows the compressibility and green strength.

Mix	Hall Flow (s/50g)	Apparent Density (g/cm³)	Green Density (g/cm³)			Green Strength (MPa)		
			500 MPa	650 MPa	800 MPa	500 MPa	650 MPa	800 Mpa
1	28.8	2.91	6.20	6.50	6.70	9.8	14.6	18.8
2	30.2	2.93	6.21	6.51	6.70	9.2	13.8	17.9
3	35.0	3.01	6.23	6.52	6.72	7.6	11.9	15.2
4	43.5	3.03	6.25	6.54	6.73	6.1	9.7	12.9
5	44.2	2.73	6.14	6.43	6.63	10.6	15.0	18.2
6	40.0	3.00	6.24	6.52	6.71	6.8	10.2	13.1

Table III. Hall Flow, Apparent Density, Green Density and Green Strength for the mixes.

The Amide wax mix has very poor Hall flow value. However the Kenolube^{TM} mix is the only mix with a Hall flow less than 30 seconds. In Kenolube^{TM} /Li-stearate mixtures, higher Lithium stearate ratio gives higher flow values. A key factor for this behaviour is the influence of the particle size distribution of these lubricants. The AD level also depends on the lubricant choice, where Lithium stearate giving the highest level. Amide wax® gives very low AD compared to the other two lubricants.

Regarding compressibility, both Kenolube^{TM} and Lithium stearate give high values, Amide wax gives approximately 0,1 g/cm³ less than the other two. Looking on the green strength, Kenolube^{TM} and Amide wax performs much better than Lithium stearate. Adding Lithium stearate to the other two lubricants has a negative effect on the green strength. The combinations give compressibility that could be predicted when knowing the result from the pure lubricants.



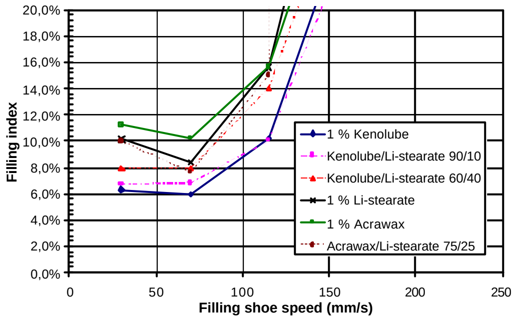


Figure 3. Die filling simulator results versus cavity volume for mix number 2.

Figure 4. Filling index plotted versus filling shoe speed for all the mixes.

Figure 3 shows the die filling simulator test result for mix number 2. The curves represent different filling shoe speeds, where the highest speed apparently gives the lowest filling density. In the diagram there are both filled and open markers for the data points. The open markers represent cavities that were not completely filled in the test. The filled markers show vales from completely filled cavities, however the density varies with both the cavity volume and the filling shoe speed. This is very important to know when producing high-density components with high shrinkage. A poor density distribution in the green compact will affect the shrinkage during sintering and as a result of this also the tolerances of the finished product.

The Kenolube^{TM} mix shows the best filling index over the whole filling shoe rate range, which means that the dependence of cavity volume is the lowest in this test. Amide wax and Li-stearate show the worst filling index. Adding Lithium stearate to Kenolube^{TM} influences the filling index slightly negative, but small additions are acceptable. All mixes have an upper limit for the filling speed at approximately 80 mm/s. Exceeding this filling speed, some of the smallest cavities were not completely filled and because of this the filling index apparently gets very high.

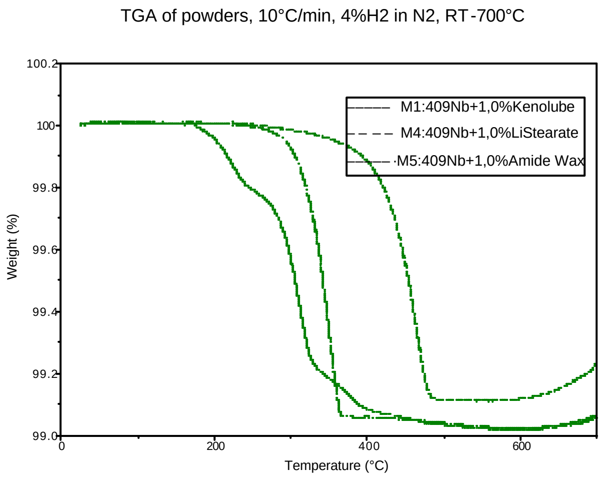


Figure 5. TGA curves of mixes 1, 4 and 5.

The TGA shows that the Amide wax and Kenolube has the cleanest burn off characteristics, with only traces of the lubricant remaining after heating to 570°C. Li-stearate however shows higher residue amount after dewaxing. The lubricants have different dewaxing temperatures; Amide wax has the lowest and Li-stearate the highest. Kenolube has the largest dewaxing temperature interval, starting already at 200°C.

Mix #	**Dimensional change (%)**			**Sintered density (g/cm^3)**		
	500 MPa	650 MPa	800 MPa	500 MPa	650 MPa	800 MPa
1	-5.3	-4.5	-3.9	7.16	7.32	7.41
2	-5.5	-4.7	-4.0	7.20	7.35	7.45
3	-5.6	-4.7	-4.0	7.24	7.37	7.45
4	-5.6	-4.7	-4.1	7.25	7.38	7.49
5	-5.6	-4.6	-4.0	7.16	7.27	7.36
6	-5.6	-4.8	-4.1	7.26	7.40	7.47

Table V. Dimensional change and density after sintering 2462°F (1350 °C), 30 minutes.

The sintered densities for the highest compaction pressure vary from 7.36 to 7.49 g/cm^3 which is interesting in view of the fact that the only difference is the lubricant choice. The density is clearly increased by Lithium stearate addition to the mixes. It increases not only the compressibility, but also the sintering activity expressed as dimensional change. Only 0.25 % addition to the Amide wax mix gives more than 0.1 g/cm^3 increase in sintered density compared to Amide wax. Furthermore, although mix #2 only has 0.1 % Lithium stearate, the sintering activity is improved compared to the Kenolube^{TM} mix.

Lubricant	**UTS (MPa)**			**Elongation (%)**			**Impact energy (J)**		
	500 MPa	650 MPa	800 MPa	500 MPa	650 MPa	800 MPa	500 MPa	650 MPa	800 MPa
Kenolube	356	367	383	14,2	10,9	11,2	145	200	224
Kenolube/Li-stearate 90/10	361	383	397	12,5	11,9	12,4	169	237	267
Kenolube/Li-stearate 60/40	367	381	400	12,2	12,5	13,1	189	278	>300
Li-stearate	371	388	412	11,9	13,4	13,4	212	>300	>300
Acrawax	357	362	373	13,0	11,3	12,0	167	185	226
Acrawax/Li-stearate 75/25	369	391	402	12,0	12,9	13,5	204	281	>300

Table VI. Tensile and impact properties after 1350 °C sintering

The mechanical properties are strongly influenced by the choice of lubricant. Lithium stearate gives a very active sintering with strong sintering necks. The UTS is as expected depending on the sintered density, but it is also affected by the lubricant choice. The strength of all mixes containing Li-stearate are higher compared to the Kenolube^{TM} and Amide wax mixes, even when compared at the same density. Amide wax and Kenolube^{TM} seem to give the same strength at the same density level. Lithium stearate mixes have the highest impact strength. The impact energy test equipment has an upper limit of 300 J, this is the reason why some of the samples could not be tested.

Discussion

Not only the powder properties depend on the lubricant used but also the final density, tolerances, mechanical and corrosion properties. When making high performance P/M stainless steel components, it is today necessary to sinter with high shrinkage. If the density distribution in the green compact is uneven, the shrinkage will be different in different areas of the component with distortion of the sintered part as a result. Hall flow and AD are often used as a quality control instrument. However these methods do not predict filling performance, this has been reported before[1, 2]. The die filling simulator used in this experimental approach can predict the behaviour in production environment. Therefore it is possible to rank different mixes from a performance point of view.

Kenolube^{TM} facilitates excellent powder properties when added to stainless steel powder mixes. Kenolube^{TM} provides fast flow, good fillability, relatively high compressibility, and excellent green strength. The reason for this is partly the particle size distribution, which is 27 µm in average. The dewaxing properties are very good compared with Li-stearate.

Amide wax has almost the same average particle size as Kenolube^{TM}, however most powder properties are inferior to Kenolube^{TM}. This lubricant gives less compressibility, almost no flow and poor performance in the die-filling simulator. However it gives very high green strength. The sintered density will be very low compared to the other lubricants tested. The lubricant removal is similar to Kenolube.

Lithium stearate gives the least favourable powder properties. The flow and fillability are not good, especially when compared to Kenolube^{TM}. However, the sintering activity of mixes lubricated with Li stearate is extremely high. The compressibility and sintered density are very high and the sintering necks are exceptionally strong, which was registered in the tensile and impact strength. The dewaxing is not complete at 570 °C and this is perhaps partly the reason behind the good sintered properties.

All lubricants have their advantages and drawbacks; this is why the mixes with combined lubricants were included in the test plan. Amide wax and Lithium stearate mixed in the relative proportion 75/25 give very good sintered properties. However, the powder properties such as flow, fillability and green strength are not very good. In this respect it would be better to combine Kenolube^{TM} with Lithium stearate. By doing this the best combination of properties can be achieved.

Conclusions

- Kenolube^{TM} gives the overall best properties when flow, fillability, and green strength are taken into account. The sintering activity and mechanical properties after sintering are equal to Amide wax but lower than Li-stearate.
- Lithium stearate gives the best compressibility, sintering activity and mechanical properties. The powder properties are not as good as for the other two lubricants in this study
- Amide wax has similar dewaxing properties as Kenolube. On all other properties tested it is inferior to the other two lubricants
- Combining the lubricants gives very interesting properties, were a mixtures of Kenolube^{TM} and Lithium stearate have the overall best performance.

References

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