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Higher Densities of PM-Steels by Warm Secondary Compaction and Sizing

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Abstract

Reaching higher densities is one of the biggest challenges for PM products today. This paper presents how new compaction processes like secondary pressing and sizing at elevated temperatures increase density and mechanical properties of sintered steels.
A prealloyed iron powder Astaloy CrL with 1.5 % Cr and 0.2 % Mo (Höganäs AB) was used without and with an amount of 0.5 % graphite. The powders were compacted by different methods that combine double press/double sinter, warm compaction for both pressing stages and warm sizing. The compaction and sizing temperatures were ranged from 20 °C to 200 °C. The various processing routes are compared through measurements of density, microstructure and mechanical properties. The results show that elevated temperatures in different compaction stages lead to a significant increase of the relevant properties.

Keywords: increased density, warm compaction, warm secondary pressing, warm sizing, low-alloyed PM steels

Introduction

Advantages of powder metallurgy like high utilization of material, the possibility to produce compacts with complex geometries in large scale, high precision and lower costs are factors in competition with alternative processing routes [1, 2]. An important objective is to produce precision parts with improved material properties through new alloying concepts or higher densities by modified processing routes. In the last years, developments showed that warm compaction leads to a significant increase in density and thereby improves the material properties [3 - 6]. The increased temperature during the compaction process results in a reduction of the yield strength of the powder particles [6, 7] which leads to a lower resistance against plastic deformation during compaction. Thus, higher density levels can be reached without increasing the pressure [7]. A well known way to increase density is the double press/double sinter method (DP/DS). In the first sintering step at moderate temperature levels, the lubricant in the powder mixture is removed [8-10]. During second pressing, the compact is densified further than in the single pressed/single sintered state (SP/SS) because the powder particles are softened during the first sintering and can be deformed more easily in the second compaction step. Furthermore, the pore space that was occupied by lubricant can now be filled with iron-based material [8].

This paper analyses to what extent combinations of these different processing routes can enhance the density of sintered steels. In a first approach the effect of reducing the yield strength of the material by using increased temperatures was applied to the sizing operation of the sintered components. Therefore, powder mixtures of prealloyed Astaloy CrL-powder (Höganäs AB) with 0 % C and one with 0.5 % C were single pressed and sintered. The temperature during sizing was varied between room temperature (RT) and 200 °C. In further experiments also the compaction temperature was varied in the same range for SP/SS and DP/DP method to identify the effect on density and properties of elevated temperatures in each processing stage.

Experimental

Prealloyed iron-based powder Astaloy CrL with 1.5 % Cr and 0.2 % Mo was used in this study. The Astaloy CrL powder was mixed with 0.8 % lubricant (Deurex®) for cold compaction (series (A)). In series (B) a graphite amount of 0.5 % was admixed. Powders were mixed in a turbular mixer for 20 min.

Only for warm compaction no lubricant was added to the powder mixture. To decrease the friction between powder particles and the die wall in this case, a suspension of temperature-resistant oil and MoS_2 was used for die wall lubrication.

A special warm compacting and warm sizing tool for tensile specimens (DIN EN ISO 2740) was designed and produced in cooperation with Schunk GmbH, Germany, and Alvier AG, Switzerland, for a hydraulic press Lauffer HPM 60LS. The die is heated by eight and the upper punch by two cartridge heaters. The lower punch is heated indirectly through the die. The temperature is measured with thermocouples in the die and in the upper punch and can be varied from RT to 200 °C. To avoid temperature transmission from the die and the punch to the tool axes, the die shrink ring and the punch plates are water cooled.

The powders were pressed under different conditions as shown in Figure 1.

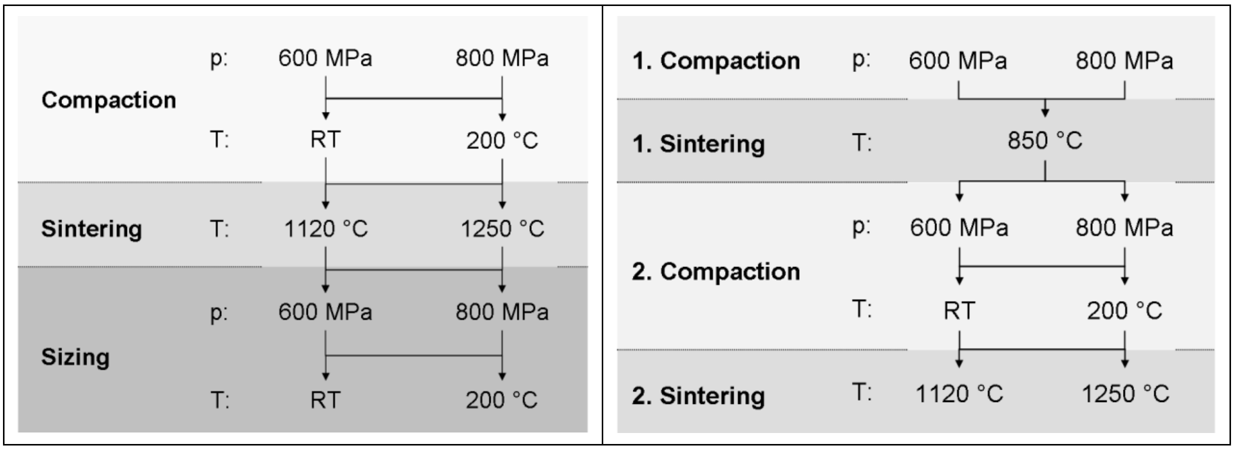

Figure 1: Schematic processing route of SP/SS (left) and DP/DS (right) with different processing parameters (p: pressure, T: temperature)

For the SP/SS-processing route (Figure 1, left) samples were compacted with varied compaction pressure (600 MPa / 800 MPa) and temperature (RT / 200 °C). Green compacts were sintered for 30 min in a tube furnace at 1120 °C respectively 1250 °C under N_2/H_2 (90/10) atmosphere and furnace cooled. The same pressures and temperatures were applied in the sizing operation of the samples.

For the DP/DS-processing route (Figure 1, right) the first compaction step was carried out at 600 MPa and 800 MPa at RT. The compacts were pre-sintered at 850 °C for 30 min in N_2/H_2 (90/10). Afterwards the pre-forms were recompacted with varied pressure (600 MPa / 800 MPa) and temperature (RT / 200 °C). All samples were then sintered at 1120 °C or 1250 °C for 30 min under N_2/H_2 (90/10) atmosphere. The DP/DS-samples were not finally sized after sintering.

To compare the material properties as a function of the processing route, densities, microstructures, tensile strengths and elongations were analysed. Green density, sinter density, and density after sizing were measured geometrically. All results that are reported are an average of at least 3-5 samples for every processing route.

Results and discussion

The results of the SP/SS-experiments without sizing and in comparison after the sizing step are presented in Table 1 and Table 2 respectively.

Table 1: Tensile strength R_m and elongation A for Astaloy CrL (A) and Astaloy CrL + 0.5 C (B) after SP/SS without sizing for different compaction pressures and sintering temperatures

Processing Parameters		Astaloy CrL		Astaloy CrL + 0.5 C	
Compaction	Sintering	R_m MPa	A %	R_m MPa	A %
600 MPa	1120 °C	131	0.6	341	0.7
	1250 °C	224	1.5	412	3.5
800 MPa	1120 °C	134	0.5	415	1.9
	1250 °C	247	2.8	495	3.5

Table 2: Density, tensile strength R_m and elongation A for Astaloy CrL (A) and Astaloy CrL + 0.5 C (B) after SP/SS with sizing for different processing parameters

					Astaloy CrL					Astaloy CrL + 0.5 % C				
Processing Parameters					Density:			Mech. Properties:		Density:			Mech. Properties:	
Comp.		Sint.	Sizing		Comp.	Sint.	Sizing	R_m MPa	A %	Comp.	Sint.	Sizing	R_m MPa	A %
600 MPa	RT	1120 °C	600 MPa	RT	6,87	6,85	7,21	257	0,3	6,77	6,80	6,95	359	2,2
				200 °C			7,25	251	0,2			6,97	380	2,4
			800 MPa	RT			7,38	273	0,3			7,08	404	2,3
				200 °C			7,38	325	0,2			7,10	440	2,2
		1250 °C	600 MPa	RT		6,93	7,25	306	3,3		6,87	7,00	384	2,9
				200 °C			7,34	303	2,1			6,99	399	2,7
			800 MPa	RT			7,41	348	0,4			7,12	431	3,0
				200 °C			7,36	328	0,3			7,16	451	3,2
	200 °C	1120 °C	600 MPa	RT	7,16	7,10	7,40	305	1,9	7,11	7,04	7,14	415	2,8
				200 °C			7,38	297	2,1			7,16	404	2,5
			800 MPa	RT			7,44	308	2,2			7,22	416	2,0
				200 °C			7,47	338	1,7			7,23	438	2,4
		1250 °C	600 MPa	RT		7,10	7,37	310	2,3		7,06	7,17	434	2,8
				200 °C			7,34	323	3,0			7,19	436	1,9
			800 MPa	RT			7,43	332	2,8			7,24	446	2,6
				200 °C			7,46	344	1,8			7,25	437	2,2
800 MPa	RT	1120 °C	600 MPa	RT	7,14	7,09	7,39	285	1,4	7,01	7,04	7,16	432	3,0
				200 °C			7,34	307	1,6			7,12	419	2,8
			800 MPa	RT			7,48	302	0,3			7,20	432	2,7
				200 °C			7,41	266	0,2			7,22	433	2,2
		1250 °C	600 MPa	RT		7,18	7,41	308	3,4		7,07	7,17	443	3,5
				200 °C			7,24	330	1,4			7,16	440	3,6
			800 MPa	RT			7,50	345	1,9			7,20	442	2,5
				200 °C			7,42	368	1,3			7,24	463	3,0
	200 °C	1120 °C	600 MPa	RT	7,33	7,27	7,46	299	4,2	7,32	7,23	7,35	467	3,0
				200 °C			7,46	283	5,3			7,34	463	2,6
			800 MPa	RT			7,47	294	3,0			7,38	480	3,2
				200 °C			7,47	325	2,7			7,40	482	2,5
		1250 °C	600 MPa	RT		7,28	7,48	289	-		7,24	7,32	465	3,0
				200 °C			7,45	313	5,1			7,34	463	2,3
			800 MPa	RT			7,44	305	3,2			7,39	497	2,8
				200 °C			7,50	328	4,0			7,37	476	1,8

For the alloy without carbon (A) the tensile strength is increased through work hardening during sizing by approximately 100 MPa. The higher the starting density after sintering, the less increase in density can be achieved by sizing. In the alloy with 0.5 % C (B) the hardening effect due to the carbon prevents further densification of the material during sizing and the tensile strength is not enhanced.

Regarding Table 2 for both alloys (A) and (B), higher densities can be obtained by increasing compaction and sizing pressure as well as compaction and sintering temperature. The density of Astaloy CrL without C (A) is enhanced from 7.2 g/cm^3 to 7.5 g/cm^3 and with 0.5 % C (B) from 6.9 g/cm^3 to 7.4 g/cm^3. Higher densities lead also to increased mechanical properties. Tensile strength of (A) is raised from 257 MPa to 368 MPa and of (B) from 359 MPa to 497 MPa. The lower density of alloy (B) is a consequence of the carbon addition to the powder mixture. An amount of 0.5 % C lowers the relative density by 0.08 g/cm^3.

For both alloys (A) and (B) the densities are increased by higher compaction pressures. As expected, improved values for tensile strength and elongation are achieved too. The same properties can be reached for low compaction pressures if the compaction temperature is increased to 200 °C. Even higher values are obtained if high compaction pressure is combined with the elevated temperature during compaction (Table 2). In contrast, increasing the sizing temperature to 200 °C does not further enhance the density and the mechanical properties. These results contradict the expectations that elevated temperatures during both, compaction and sizing, increase the density as a consequence to the reduced yield strength of the material.

Table 3 presents the results for the DP/DS-samples of both alloys (A) and (B). The densities after first compaction, pre-sintering at 850 °C, second compaction and final sintering are listed as well as the tensile strength and elongation.

Table 3: Density, tensile strength R_m and elongation A for Astaloy CrL (A) and Astaloy CrL + 0.5 C (B) after DP/DS for different processing parameters (1. C.: first compaction, 1. S.: pre-sintering, 2. C.: recompaction, 2. S.: sintering)

				Astaloy CrL						Astaloy CrL + 0.5 % C					
Processing Parameters				Density:				Mech. Properties:		Density:				Mech. Properties:	
1. C.	1. S.	2. C.	2. S.	1. C.	1. S.	2. C.	2. S.	R_m MPa	A %	1. C.	1. S.	2. C.	2. S.	R_m MPa	A %
600 MPa	850 °C	600	1120°C	6,87	6,82	7,21	7,15	246	9,2	6,86	6,77	7,16	7,13	401	2,5
		RT	1250°C				7,27	281	17,1				7,27	428	3,3
		600	1120°C			7,33	7,23	287	13,0			7,24	7,22	502	5,6
		200°C	1250°C				7,30	275	22,7				7,31	495	6,5
		800	1120°C			7,34	7,23	254	10,0			7,28	7,25	460	3,9
		RT	1250°C				7,37	287	21,8				7,31	480	4,4
		800	1120°C			7,34	7,34	296	22,0			7,32	7,29	530	5,6
		200°C	1250°C				7,41	305	24,6				7,35	545	6,8
800 MPa	850 °C	600	1120°C	7,09	7,02	7,28	7,22	254	10,0	6,95	6,93	7,23	7,32	485	4,6
		RT	1250°C				7,28	284	17,9				7,21	551	4,1
		600	1120°C			7,38	7,36	274	21,6			7,26	7,26	495	4,6
		200°C	1250°C				7,38	278	27,2				7,38	534	7,7
		800	1120°C			7,40	7,32	283	15,8			7,30	7,39	490	4,2
		RT	1250°C				7,42	316	20,4				7,29	528	6,8
		800	1120°C			7,41	7,35	301	19,7			7,35	7,31	539	5,1
		200°C	1250°C				7,45	289	26,0				7,34	586	6,5

For DP/DS, as expected, in the first compaction increased pressure leads also to higher densities. After pre-sintering at 850 °C the density is slightly decreased. Since no dimensional changes of the compact are observed during pre-sintering the density change can easily be explained by the removal of the lubricant. The pre-sintering temperature of 850 °C was chosen to reach the complete annealing of work hardening effects during the first compaction. To prevent the diffusion of carbon contained in the mixture of alloy (B) the pre-sintering temperature has to be as low as possible. As the density increase of the alloy (B) after recompaction shows, the choice of 850 °C as pre-sintering temperature represents a good compromise. It should be pointed out, that the removal of the lubricant creates better pre-conditions for further densification than to the standard warm compaction process.

Using standard pressures of 600 MPa for the first compaction and the recompaction increases the density for the alloy (A) from 6.87 g/cm^3 to 7.21 g/cm^3. Density up to 7.33 g/cm^3 is achieved if recompaction is performed at 200 °C. A higher pressure of 800 MPa for the recompation does not result in further improvements even if the recompaction temperature is raised to 200 °C. Though an increase of the pressure in the first compaction to 800 MPa results in higher green-densities, this is

not reflected in an equivalent increase of the densitiy after recompaction (7.28 g/cm^3). Higher densities of 7.38 g/cm^3 to 7.41 g/cm^3 are obtained if high compaction pressures (800 MPa) and/or warm secondary compaction are applied.
For Astaloy CrL + 0.5 % C (B) no significant influence of the carbon content on the densities during the first compaction can be detected. As show the densities after the secondary compaction, almost no influence of carbon diffusion into the iron during pre-sintering can be found. This results in densities after recompaction that are only slightly lower than for alloy (A). The same tendencies as discussed for alloy (A) are seen for alloy (B) (Table 3).

Due to the high densities after recompaction almost no shrinkage during sintering even at 1250 °C occurs. Therefore the mechanical properties are mainly influenced by the density after the secondary compaction. For the alloy (A) the tensile strength varies in the narrow range between 246 MPa and 316 MPa. A bigger effect of the sintering temperature is caused on elongation. With the sintering temperature of 1250 °C elongations up to 27.2 % are achieved. For the alloy (B) the standard DP/DS-procedure (600 MPa / 850 °C / 600 MPa/RT / 1120 °C) results in tensile strength of 401 MPa and 2.5 % elongation. Increasing the sintering temperature to 1250 °C only slightly improves the properties. For this alloy higher densities have a significant effect on the mechanical properties. Up to 586 MPa tensile strength and 6.5 % elongation can be reached at a density of 7.34 g/cm^3 if extreme conditions are applied (800 MPa / 850 °C / 800 MPa/200 °C / 1250 °C). It should be emphasised that it is possible to increase strength by appr. 100 MPa and elongation to 5.6 % if only the temperature of recompaction is raised to 200 °C under 600 MPa / 850 °C / 600 MPa/200 °C / 1120 °C.

Conclusions

As known from the development of warm compaction the decreasing yield strength, with increasing temperature can be used to obtain higher densities. In this study the influence of elevated temperature (200 °C) in all compaction steps (first compaction, secondary compaction and sizing) was evaluated. A significant increase of density can be reached for both alloys (A) and (B) by warm compaction. Although densities in general can be increased during sizing, there was no effect of the sizing. It should be mentioned, that the tensile strength after sizing is improved due to work hardening.
The highest densities and best properties are obtained by the DP/DS-method if the secondary compaction is performed at 200 °C. Compared to the standard warm compaction this route has the advantage that no lubricant that hinders densification remained in the parts after pre-sintering.

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