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EXPERIMENTAL RESEARCH ON THE MICROSTRUCTURE AND PROPERTIES OF SOME FUNCTIONAL MODELS CREATED BY POWDER AGGREGATION IN AN AUSTENITIC STAINLESS STEEL AND ALUMINA SYSTEM

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ABSTRACT: This paper sets out to present experimental research regarding the processing of industrial applications of some bush type sintered parts (valve seats for automobiles) created from a mix of austenitic stainless steel AISI 316L with a contents of 10, 20 and 30% Al_2O_3. The set of 12 samples was exposed – after homogenizing, when 0.5 zinc stearate was added – to pressing in a pressing mold with a two column guidance and with modular elements: 6 samples were pressed at 400 kN force and 6 samples at 200 kN. After sintering at 1200°C for 1 hour in H_2 atmosphere, from each batch, 3 samples were austenitized at 1100°C for 1 hour. Metallographic microstructural analysis was done which highlighted austenitic structures with pores situated at the limits of irregular shape sintered particles and, respectively, the added component in proportional volumes to those in the primary mix. The austenitizing thermal treatment did not significantly modify the microstructural characteristics. Micro hardness determinations did not highlight significant differences between austenitic particles in different samples. Corrosion tests in acid environments (5% nitric acid and 5% sulfuric acid solutions) for 48 hours have demonstrated that corrosion effects are practically negligible. In the case of abrasive wear tests, we have noticed that an increase in alumina has resulted in lower wear. For creating functional models we recommend a mix of 316L stainless steel with 30% Al_2O_3.
KEY WORDS: powder metallurgy, stainless steel, alumina, sintered, experimental

1. INTRODUCTION

The considered metallic powder was based on AISI 316L stainless steel. The chemical composition (% mass) of the used powder was: 0.024% C; 16.78% Cr; 13.48% Ni; 2.0% Mo; 0.11% Mn; 0.77% Si; 0.1% S, the rest Fe.
The granulation of the used powder (irregular nonspherical powder) had the following values: < 40μm – 60%; 40-64μm – 18%; 63-80μm – 12%; 80-100μm – 10%.
The powder was preprocessed using 0.5% Zn stearate and, respectively, 10, 20 or 30% Al_2O_3 creating ring pieces (valve seats for automobiles, Figure 1) with the composition and dimensions presented in Table 1, at pressing forces of 400 and 200 kN. Sintering was done at SINTEROM in Cluj-Napoca, in an H_2 atmosphere at 1200°C for 1 hour and cooling in still air.

Figure 1. Research samples

We note the fact that after sintering, two samples had macro structural defects (Figure 2), determined by a faulty pressing in the matrix. These samples were removed from further research.

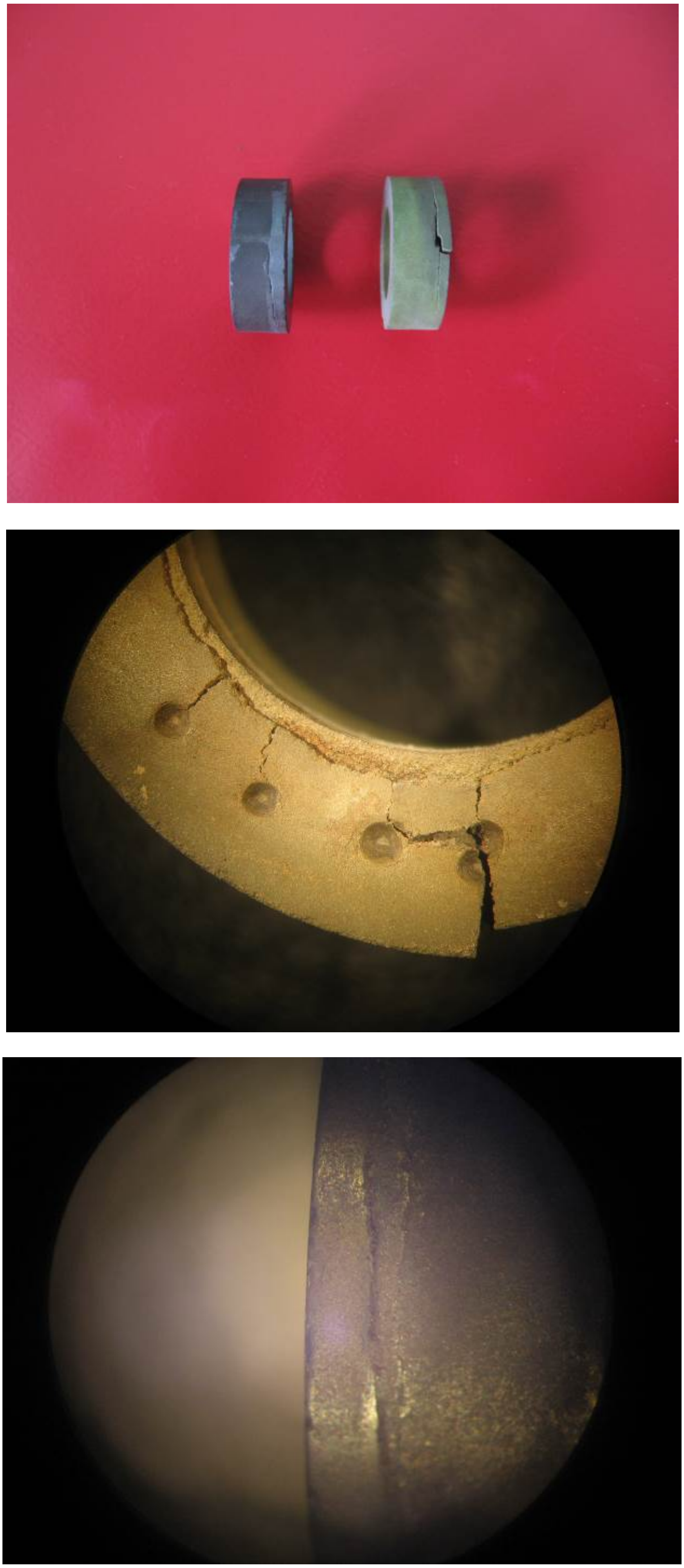


Figure 2. Images of faulty samples

Table 1. Composition and dimensions of sintered parts from 316L steel and Al_2O_3

Pressing force: 400 kN

Components		A1	A2	A3
10% Al2O3 0,5% Zn stearate	Int. Diam. (∅)	21	21	21
	Ext. Diam. (∅)	34,18	34,18	34,18
	Height (mm)	10,37	10,31	10,34
	Weight (g)	39,438	39,155	39,302
20% Al2O3 0,5% Zn stearate	Int. Diam. (∅)	21	21	21
	Ext. Diam. (∅)	34,20	34,20	34,20
	Height (mm)	11,22	11,16	11,17
	Weight (g)	40,802	40,741	40,752
30% Al2O3 0,5% Zn stearate	Int. Diam. (∅)	21,03	21,04	21,03
	Ext. Diam. (∅)	34,24	34,24	34,24
	Height (mm)	11,48	11,43	11,41
	Weight (g)	38,133	38,108	37,853

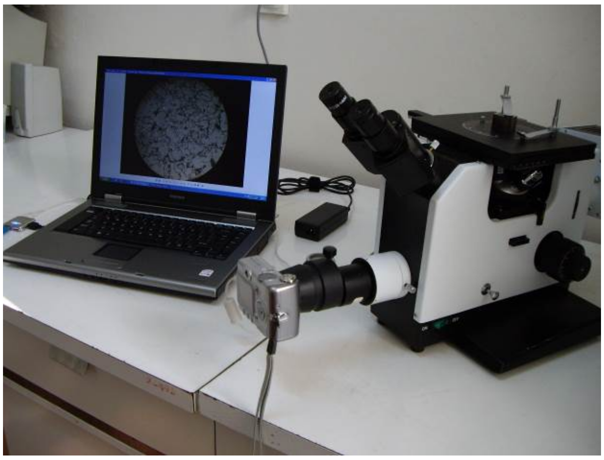
Pressing force: 200 kN

Components		A1	A2	A3
10% Al2O3 0,5% Zn stearate	Int. Diam. (∅)	21,01	21,01	21,01
	Ext. Diam. (∅)	34,15	34,15	34,15
	Height (mm)	10,94	11,03	11,79
	Weight (g)	38,868	39,433	40,472
20% Al2O3 0,5% Zn stearate	Int. Diam. (∅)	21,03	21,01	21,01
	Ext. Diam. (∅)	34,16	34,16	34,16
	Height (mm)	11,73	11,77	11,79
	Weight (g)	40,535	40,514	40,472
30% Al2O3 0,5% Zn stearate	Int. Diam. (∅)	21,03	21,04	21,04
	Ext. Diam. (∅)	34,19	34,19	34,18
	Height (mm)	12,07	12,01	12,00
	Weight (g)	39,391	37,990	38,149

2. MICROSTRUCTURAL METALLOGRAPHIC ANALYSIS

Microscopic metallographic analysis was done on a NEOPHOT 21 / C. Zeiss microscope and the photographing of microstructures was done on a metallographic microscope KRUSS MMB 2200 with a Canon A520 digital camera (Figure 3).

a.



b.

c.

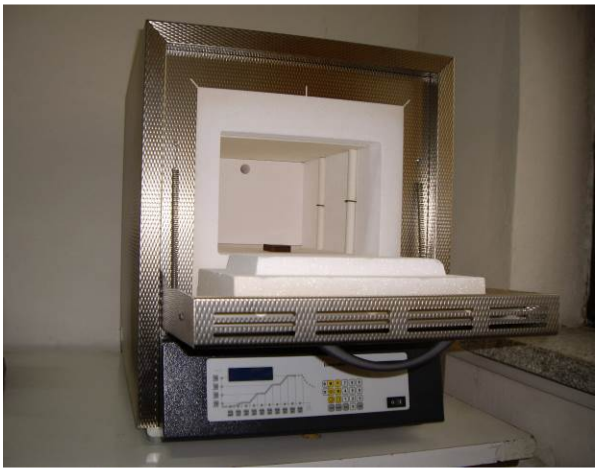
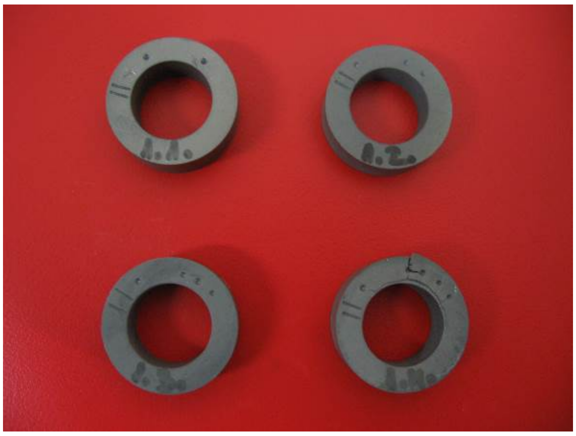

d.

Figure 3. Investigation means used for research

a – NEOPHOT 2.1 – C. Zeiss microscope; b – KRUSS MMB 2200 microscope; c – CV – 400 DAT – NAMICON digital hardness tester; d – L 15/11 NABERTHERM oven for thermic treatments

Samples for microstructural analyses were polished with classical procedures, then the metallographic attack was commenced with a reactive with the following composition: 10 ml HNO_3; 20 ml HCl; 30 ml distilled water.

Figure 4 and Table 2 show the samples considered for metallographic analysis and Figures 5, 6, 7 and 8 present their microstructures.


a.


b.


c.


d.

Figure 4. Images of sintered samples

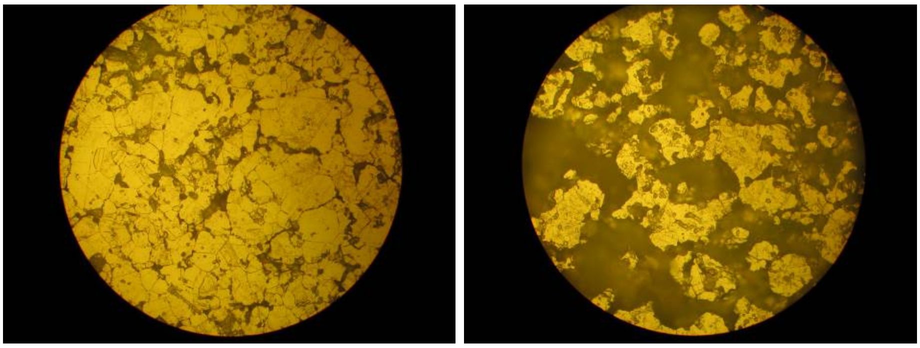
a – P type samples; b – P_t type samples; c – D type samples; d – D_t type samples

Microstructural analysis, done at 500x magnification, highlights a typical austenitic structure with pores at the edges of irregular shaped sintered particles and, respectively, added compound in proportional volumes to those in the primary mix. The eventual effects of a supplementary austenitic treatment 1100°C/1h on some samples (noted with P_t and D_t) were analyzed. This treatment was done in a Nebertherm L15/11 oven with programmable cycles and electronic control (Figure 3 d).

The austenitic treatment applied to P_t and D_t type samples has not significantly modified the microstructural characteristics, determining just an accentuation of austenitic microstructural characteristics of 316L steel particles, while maintaining a constant porosity and relative quantity of added component determined in P and D type samples. Microstructural analyses did not reveal noticeable quantitative (volume) differences between samples pressed at 400 kN compared to those pressed at 200 kN.

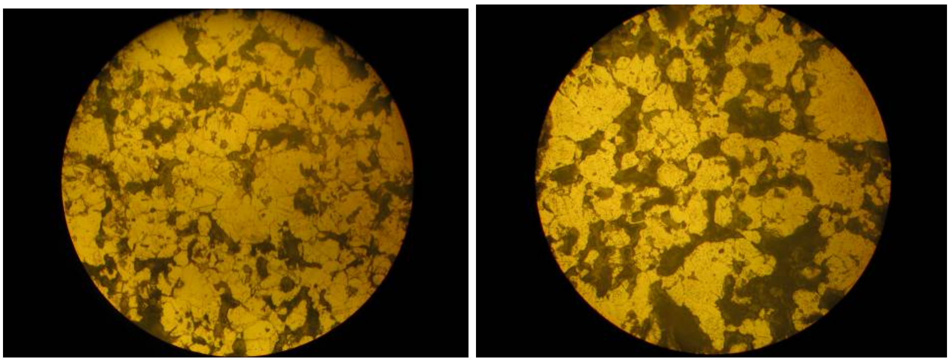
Table 2. Symbols and characteristics of sintered 316L steel samples

Symbol	Components	Pressing force [kN]	Treatment
P1.2	10% Al2O3 0,5% Zn stearate	400	No
P1.3	20% Al2O3 0,5% Zn stearate	400	No
P1.4	30% Al2O3 0,5% Zn stearate	400	No
P_t 1.2	10% Al2O3 0,5% Zn stearate	400	Yes
P_t 1.3	20% Al2O3 0,5% Zn stearate	400	Yes
P_t 1.4	30% Al2O3 0,5% Zn stearate	400	Yes
D1.2	10% Al2O3 0,5% Zn stearate	200	No
D1.3	20% Al2O3 0,5% Zn stearate	200	No
D1.4	30% Al2O3 0,5% Zn stearate	200	No
D_t 1.2	10% Al2O3 0,5% Zn stearate	200	Yes
D_t 1.3	20% Al2O3 0,5% Zn stearate	200	Yes
D_t 1.4	30% Al2O3 0,5% Zn stearate	200	Yes



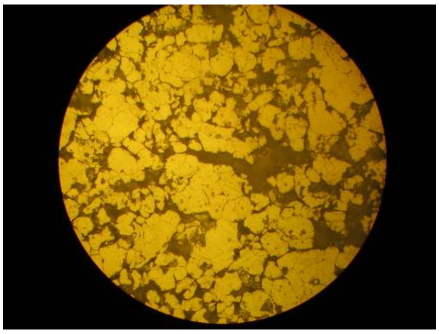
a. b. c.

Figure 5. Microstructures of sintered samples, 400kN (x500) a – P 1.2; b – P 1.3; c – P 1.4



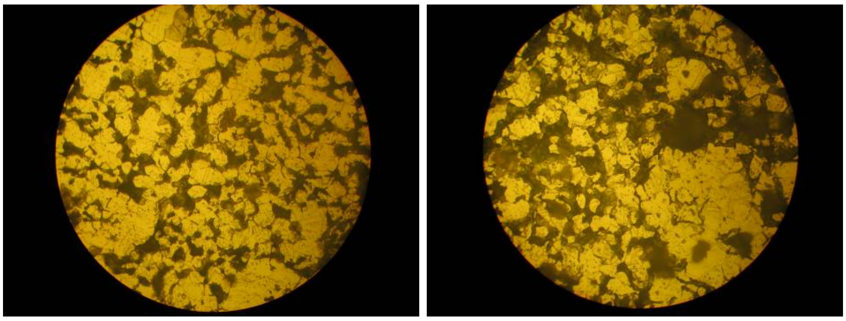
a. b. c.

Figure 6. Microstructures of sintered samples, 400kN (x500) a – P_t 1.2; b – P_t 1.3; c – P_t 1.4



a. b. c.

Figure 7. Microstructures of sintered samples, 200kN (x500) a – D 1.2; b – D 1.3; c – D 1.4



a. b. c.

Figure 8. Microstructures of sintered samples, 200kN (x500) a – D_t 1.2; b – D_t 1.3; c – D_t 1.4

3. MICRO HARDNESS AND HARDNESS DETERMINATIONS

Micro hardness (HV0,1) determinations targeted component particles from austenitic steel and were done both with a CV-400 DAT/NAMICON digital hardness tester as well as with and mph – C. Zeiss micro harness tester mounted on a NEOPHOT 21/C. Zeiss metallographic microscope. The obtained values (Table 3) did not show significant differences between the micro hardness of austenitic particles in different samples.

Brinell (ϕ2,5/187,5 kgf) hardness tests have shown that pressing at 200 kN has yielded a 10-20% lower hardness of sintered samples compared to the values for pressing at 400 kN, without any noticeable difference for the supplementary austenitic treatment.

Correlating these observations with microstructural analyses of samples, we consider that HB hardness variations for introducing 10, 20 or 30% Al_2O_3 did not present significant differences, these variations being determined by the inherent inhomogeneity of the composition of sintered mixes.

Table 3. Values for HV0,1 micro hardness and HB hardness

Symbol	HV 0,1	HB
P1.2	270	177
P1.3	266	156
P1.4	258	175
Pt 1.2	251	153
Pt 1.3	256	174
Pt 1.4	264	153
D1.2	262	130
D1.3	258	128
D1.4	260	131
Dt 1.2	254	128
Dt 1.3	264	122
Dt 1.4	247	130

4. CORROSION DETERMINATIONS IN ACID EVIRONMENTS

Samples (ϕ 21 x 12.5 mm) were tested for corrosion in weak acid environments (5% nitric acid and 5% sulfuric acid solutions). Samples were immersed for 48h in the aforementioned solutions, with results shown in Table 4.

Even though some small weight differences were noticed, as a result of developed chemical reactions, macro structural analyses done on samples (Figure 9) have shown that corrosive effects are negligible in such environments, the surface aspect of the samples was also not affected (limited by some very light local loss in the initial polish on samples that were previously polished or some light local coloring, extremely superficial and which could be removed by simply wiping with a soft cloth).

a. b. c.

Figure 9. Macrostructure of sintered stainless steel samples, tried for corrosion in acid environments

a – D 1.4 (5% H_2SO_4 solution); b – D_t 1.2 (5% HNO_3 solution); c – D_t 1.4 (5% HNO_3 solution)

Table 4. Results obtained by attacking samples (immertion) in 5% HNO_3 and 5% H_2SO_4 solutions

Sample type	Acid attack [5%]	Initial weight, [g]	Weight after 48 h, [g]
D1.2	HNO3	20,943	21,011
D1.3	HNO3	21,316	21,338
D1.4	HNO3	20,010	19,979
D1.2	H2SO4	20,602	20,691
D1.3	H2SO4	20,998	21,064
D1.4	H2SO4	19,589	19,634
Dt1.2	HNO3	20,337	20,378
Dt1.3	HNO3	20,858	20,942
Dt1.4	HNO3	18,792	18,773

Note: After immersion in acid solution, the samples were dried on filter paper and then heated in an oven at 100°C for 10 minutes followed by a cooling to ambient temperature for one hour.

5. ABRASIVE WEAR TRIALS

These trials were done on P type samples in the following conditions: Contact type: pine/disc; Abrasive paper SR9795/3/94 type PEZ522X, gr. 60; Contact pressure: 0.7 MPa; Radial advance: 0.45 mm/rot; Sliding distance: 71.2 m; Rotation speed: 25 rot/min.

Weight loss was as follows:

Sample type	P 1.2 (10 % Al2O3)	P 1.3 (20 % Al2O3)	P 1.4 (30 % Al2O3)
Weight loss (g)	1,78	1,24	0,98

We notice that increasing the Al_2O_3 quantity resulted in a reduced wear, the correlation of the wear with the Al_2O_3 quantity is presented in Figure 10

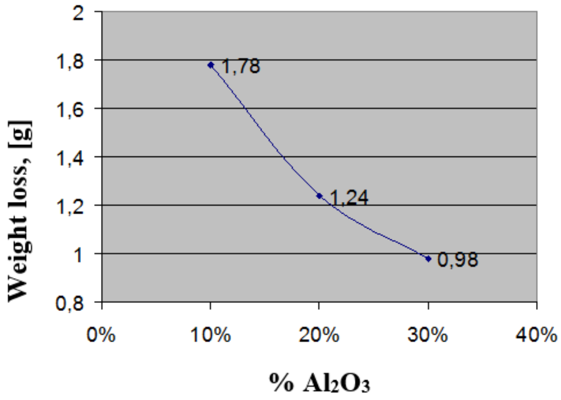


Figure 10. Wear of P type samples

6. CONCLUSIONS

- From a microstructural point of view, increasing the alumina quantity is clearly visible; the eventual supplementary austenitic treatment has slightly highlighted the austenitic characteristics of respective particles, while maintaining porosity and proportions of mix components;
- HB hardness of samples pressed at 400 kN is 10-20% higher than that of samples pressed at 200 kN;
- Hardness of samples shows no difference in correlation with the introduced Al_2O_3 quantity;
- The supplementary 1100°C/1h austenitic treatment does not determine visible modifications of micro hardness or hardness;
- Resistance to abrasive wear of type pine/disc is higher with higher quantities of alumina introduced in the mix;
- Sintered samples present a good resistance to corrosion in solutions of HNO_3 and H_2SO_4 but they are attacked by saline fog, without a certain dependence of corrosion resistance to the quantity of Al_2O_3;
- For creating the functional models we recommend a mix of 316L stainless steel with 30% Al_2O_3, pressed at 400 kN force.

7. REFERENCES

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