

# Research on the surface densification of sintered steels

A. CALOPEREANU, C. NICOLICESCU\*, V. NICOARĂ

University of Craiova, Faculty of Mechanics, Department of Engineering and Management of the Technological Systems, Drobeta Turnu Severin, Romania

Surface densification of sintered steel parts has effects in terms of increasing of the fatigue and wear resistance and is currently conducted by shocks with balls (shot peening) or rolling. This paper presents the results of a new method of surface densification of sintered steels namely densification by cold plastic deformation using planetary ball mill. Sintered steel with 0,15%C and 0,25%C were subjected to surface densification using a Pulverisette 6 planetary ball mill with three rotational speeds of 150, 250 and 350 rpm for 5, 15 and 20 minutes at each rotation. It was analyzed the effect of the densification process on the roughness, thickness and the characteristics of the densified layers respectively the behavior of the densified steels to impact tests.

(Received May 20, 2015; accepted October 28, 2015)

**Keywords:** Sintered steel, Shot peening, Porosity, Impact test

## 1. Introduction

The presence of pores in the structure of the sintered steels has negative influence on the mechanical properties. For this reason, in the case of parts made from sintered steels which are subjected to high stresses, it is necessary to practice some operations, in order to obtain higher densities and lower porosities.

After compaction and sintering can be achieved densities in the range of (6,8-7,2) g/cm<sup>3</sup>, while by the post-sintering operations as densification, can be achieved densities close to the theoretical density and thus very low porosities [1-3].

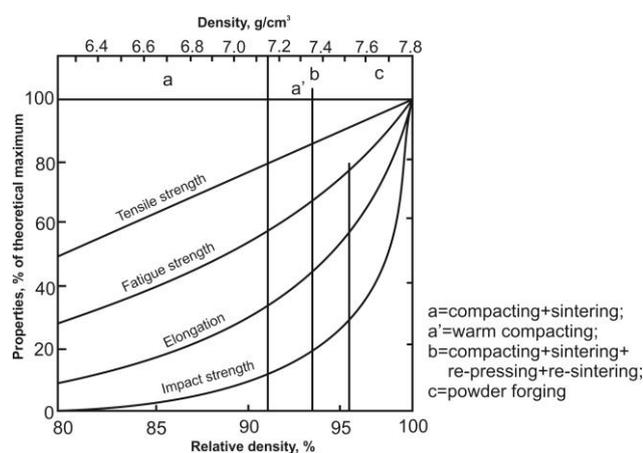


Fig. 1. Density and mechanical resistance of the sintered steels [1]

Densification of the surfaces of the sintered alloy parts of a certain thickness is a post sintering operation and at industrial level is carried out by two methods: by rolling, in the case of gears and by shot peening in the case of parts with a high complexity configuration.

The main goal of surface densification is the increasing of fatigue strength, hardness respective the wear resistance [4].

Surface densification by balls shock is a mechanical densification process of sintered steels surfaces, consisting in micron depths deformation of the steels under the action of the kinetic energy of the balls which strike the surfaces that will be densified, causing plastic deformation around which are accumulated compression residual stresses, fig. 2 [5].

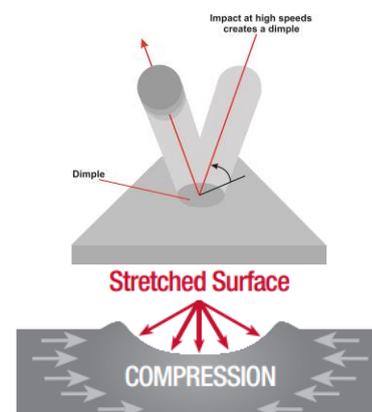


Fig. 2. Deformations that occurs in the shot peening process [5]

Table.1. Characteristics of densified sintered steels in surface [4]

Standard Reference				Properties									
DIN SINT	Core density [g/cm <sup>3</sup> ]	Surface density [g/cm <sup>3</sup> ]	ISO	Composition	Core density [g/cm <sup>3</sup> ]	Core hardness HB	Surface hardness HV <sub>01</sub>	UTS [MPa]	FEL [MPa]	YS <sub>0,1</sub> [MPa]	YS <sub>0,2</sub> [MPa]	A [%]	E [GPa]
D00	6,8-7,2	>7,6	F-00-120	Fe-0,2C	7,00	75	180	230	n/a	130	150	5	140
E00	>7,2	>7,6	F-00-140	Fe-0,2C	7,25	90	180	260	n/a	150	170	8	160
D01	6,8-7,2	>7,6	F-05-170	Fe-0,5C	7,00	90	250	300	n/a	160	180	3	140
E01	>7,2	>7,6	n/a	Fe-0,5C	7,25	120	250	340	n/a	180	210	4	160
D10	6,8-7,2	>7,6	F-00-120	Fe2Cu-0,2C	7,00	125	220	340	n/a	200	240	3	140
E10	>7,2	>7,6	F-00-140	Fe2Cu-0,2C	7,25	140	220	380	n/a	230	280	4	160
D11	6,8-7,2	>7,6	F-05C2-300	Fe2Cu-0,5C	7,00	140	300	500	n/a	300	330	2,5	140
E11	>7,2	>7,6	n/a	Fe2Cu-0,5C	7,25	180	300	570	n/a	320	360	3	160
n/a	6,8-7,2	>7,6	n/a	Fe0,85Mo-0,2C	7,00	120	260	280	n/a	160	180	4	140
n/a	>7,2	>7,6	n/a	Fe0,85Mo-0,2C	7,25	130	260	340	n/a	180	220	5	160
n/a	6,8-7,2	>7,6	n/a	Fe0,85Mo1,75Ni-0,2C	7,00	130	275	350	n/a	200	230	4	140
n/a	>7,2	>7,6	n/a	Fe0,85Mo1,75Ni-0,2C	7,25	150	275	390	n/a	220	260	4,5	160
D30	6,8-7,2	>7,6	n/a	Fe1,5Cu1,75Ni0,5Mo-0,2C	7,00	140	230	470	n/a	340	360	3,5	140
E30	>7,2	>7,6	n/a	Fe1,5Cu1,75Ni0,5Mo-0,2C	7,25	160	230	500	n/a	370	390	4	160

By this process the resistance to fatigue of the sintered steels will be increase by 70% and also the hardness in a high extent.

Industrial process consists in directing of a jet of balls on the required surfaces under the pressure of compressed air, fig. 3 [6].

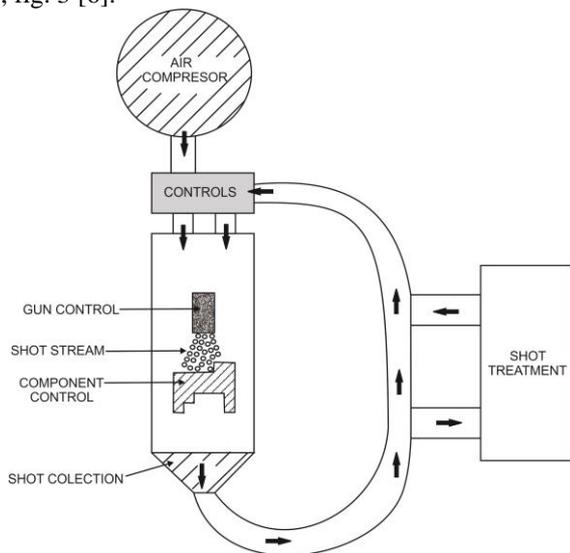


Fig.3. Air blast shoot peening equipment [6]

In the case of this equipment the gun design is very important because it control the shot stream. There are some variables in the design of the gun as following: nozzle length which can affect the energy transfer efficiency, diameter and material of the nozzle, mixing of air and shot [7-14].

The problems that appear in the densification process mentioned above are the following: the projection of the balls on the surface isn't uniform and cannot be densified curved surfaces or surfaces with complex geometry.

By these reasons was studied the application of a new process of surface densification by cold plastic deformation, the impact of the balls with high kinetic energy and surfaces that must be densified being provided by a planetary ball mill.

In this research were studied the effects of the milling parameters (rotation speeds and times) on the quality and porosities of the densified surfaces.

For the research were used sintered steels with 0,15% C and 0,25% C which were densified in a planetary ball mill Pulverisette 6 made my Fritsch. There were used three rotational speeds (150, 250 and 350 rpm) respectively three densification times for each rotational speed (5, 15 and 20 minutes).

## 2. Experimental procedure

In order to prepare the samples for the research, iron powders from Ductil SA Buzau and graphite powders were used, with the properties presented in table 2 and 3.

Table 2. Properties of iron powder

Chemical element	MAX [%]	
Carbon, C%	0,02	
Sulfur, S%	0,015	
Phosphorus, P%	0,02	
Silicon, Si%	0,05	
Manganese, Mn%	0,20	
Oxygen, O%	0,22	
Physical properties		
Property	MIN	MAX
Hall Apparent Density [g/cm <sup>3</sup> ]	2,50	2,70
Hall Flow Rate [sec/50g]	31	33
Particle size distribution		
+212 μm (%)	---	0,1
-212/+160 μm (%)	---	15
-160/+100μm (%)	20	40
-100/+63μm (%)	20	40
Pan (%)	25	45
Green properties		
Compressibility (at 600MPa-43,5tsi, 0,75% Acrawax) [g/cm <sup>3</sup> ]	6,95	---

Table 3. Properties of graphite powder

Bulk Density [g/cm <sup>3</sup> ]	1.3-1.95
Porosity [%]	0.7-53
Modulus of Elasticity [GPa]	8-15
Compressive strength [MPa]	20-200
Flexural strength [MPa]	6.9-100
Coefficient of Thermal Expansion [x10 <sup>-6</sup> °C]	1.2-8.2
Thermal conductivity [W/m.K]	25-470
Specific heat capacity [J/kg.K]	710-830
Electrical resistivity [Ω.m]	5x10 <sup>-6</sup> -30x10 <sup>-6</sup>

It was been produced two types of homogenous mixtures which consist in Fe+0,15%C and Fe+0,25%C. The two homogenous mixtures were pressed at two pressures and sintered according to the diagram presented in fig. 4 and then were surface densified using a

Pulverisette 6 planetary ball mill, all the steps being presented in table 4.

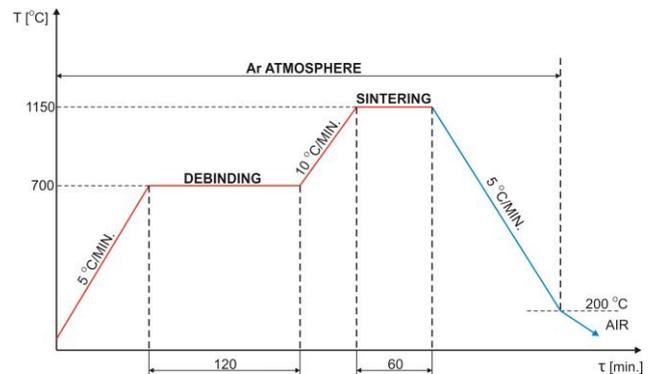


Fig.4. Sintering diagram of the steels

Table 4. Flow chart of the experiment

OPERATIONS	PARAMETERS	SAMPLES CODE
Mixing the elemental powders	Fe+0,15%C+1,5%StrZn	0,15
	Fe+0,25%C+1,5%StrZn	0,25
Compaction by die pressing	500 MPa	0,15/500
		0,25/500
	650 MPa	0,15/650
		0,25/650
Sintering	Temperature=1150 °C Dwell time=60 min. Atmosphere=argon	0,15/500-S
		0,25/500-S
		0,15/650-S
		0,25/650-S
Surface densification using Pulverisette 6 planetary ball mill	Rotational speed: 150 rot/min Times: 5, 15, 20 minute	0,15/500-150/5
		0,25/500-150/5
		0,15/500-150/15
		0,25/500-150/15
		0,15/500-150/20
		0,25/500-150/20
		0,15/650-150/5
		0,25/650-150/5
	Rotational speed: 250 rot/min Times: 5, 15, 20 minute	0,15/650-150/15
		0,25/650-150/15
		0,15/650-150/20
		0,25/650-150/20
		0,15/500-250/5
		0,25/500-250/5
	Rotational speed: 350 rot/min Times: 5, 15, 20 minute	0,15/500-250/15
		0,25/500-250/15
		0,15/500-250/20
		0,25/500-250/20
		0,15/650-250/5
		0,25/650-250/5
Rotational speed: 350 rot/min Times: 5, 15, 20 minute	0,15/650-250/15	
	0,25/650-250/15	
	0,15/650-250/20	
	0,25/650-250/20	
	0,15/500-350/5	
	0,25/500-350/5	
	0,15/500-350/15	
	0,25/500-350/15	

The green and sintered densities were measured and the sintered steels were analyzed in terms of the effects of the densification parameters on the quality respectively on the thickness, porosities and densities of the densified layer. In order to determine the density was used the relation (1) and for porosity the relation (2):

$$\rho = \frac{m}{v} \text{ [g/cm}^3\text{]} \quad (1)$$

$$P = \left(1 - \frac{d_s}{d_t}\right) \times 100 \text{ [%]} \quad (2)$$

where  $d_s$  is the sintered density and  $d_t$  is the theoretical density.

The densification process was carried out using a Pulverisette 6 planetary ball mill with the following parameters: material of the grinding vial – stainless steel, volume of the grinding vial – 250 ml, material of the grinding balls – stainless steel, diameter of the balls – 5 mm, number of densified samples – 4, ratio between the weight of the sample and weight of the balls – 1/3, rotational speeds – 150, 250 and 350 rpm, times – 5, 15 and 20 minutes.

For the microscopic analysis a Nikon microscope and NIS ELEMENTS software were used. The roughness of the surfaces which were in contact with the balls was measured using a Surtronic 25+ profilometer.

### 3. Results and discussion

The densities and the porosities of the sintered steels are presented in table 5.

Table 5. Physical characteristics of the sintered steels

Carbon content [%]	0,15		0,25	
	Characteristics			
	$\rho$	P	$\rho$	P
Pressure [MPa]				
500	6,79	12,95	6,75	13,46
650	6,88	11,79	6,84	12,31

The density of the sintered steels increases with the increasing of the compacting pressure. According to the chemical composition, the lower values of the densities are attained for the steels with 0,25% C. Being complementary with the density, the porosity is lower for the steels with higher values of the densities.

In fig. 5 are presented images with the porosity in the marginal layer.

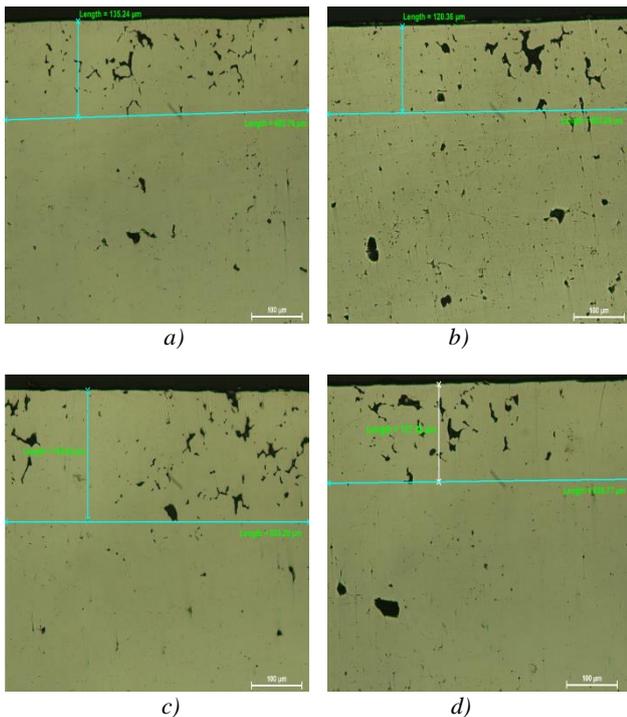


Fig. 5. Images of marginal layers of sintered steels (300X)

From the images above it is observed that the porosity is higher in the marginal layer comparative with the porosity from the core of the sintered steels.

The values of the roughness of the densified samples are presented in table 6.

Table 6. The roughness of the densified surfaces function the densification parameters

Rotational speed [rpm]	Time [min]	Carbon content [%]			
		0,15		0,25	
		Compacting pressure [MPa]			
		500	650	500	650
		Roughness Ra [ $\mu\text{m}$ ]			
150	5	0,953	1,34	1,12	1,75
	15	1,26	1,66	1,84	2,48
	20	1,44	2,64	2,31	2,98
250	5	1,06	1,37	1,16	1,6
	15	1,91	2,22	1,95	2,37
	20	2,03	2,52	2,72	2,81
350	5	1,83	2,48	2,11	2,6
	15	2,58	2,81	3,59	3,59
	20	3,79	4,03	4,56	6,88
Sintered sample		1,48	1,07	2,12	1,36

Also, in fig. 6 is revealed the quality of the densified surfaces.

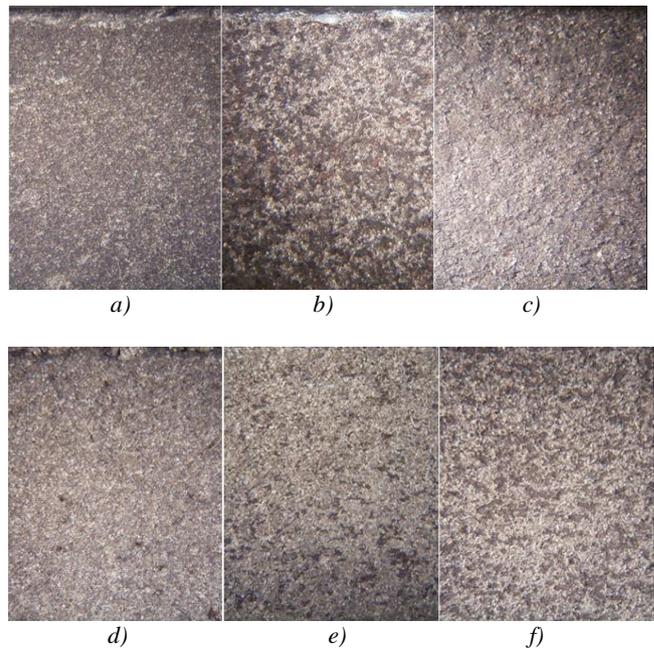


Fig. 6. Surfaces quality of the densified steels: a)0,15/650-350/5; b)0,15/650-350/15; c)0,15/650-350/20; d)0,25/650-350/5; e)0,25/650-350/15; f)0,25/650-350/20

The data from the table and fig. 6 underline the following:

- the roughness of the surfaces before deformation (sintered state) decreases with increasing of compaction pressure.

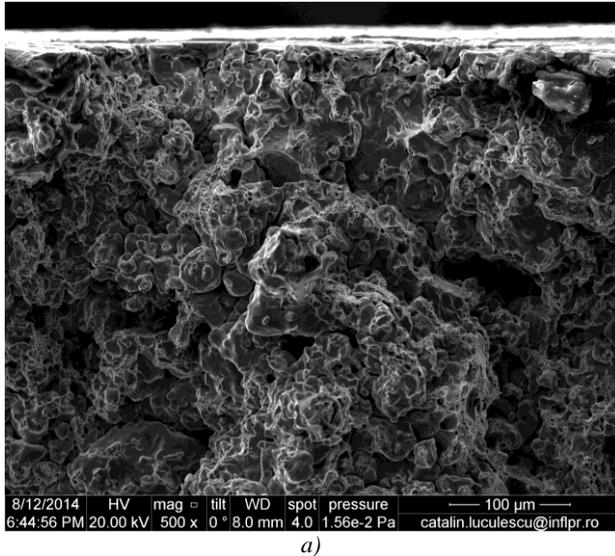
- densification by plastic deformation with rotational speed of 150 rpm of steels pressed at 500 MPa leads to surface roughness Ra lower than those of sintered samples (before deformation).

- the roughness is influenced by the milling time and it increases with the increasing of the milling time at each rotational speed;

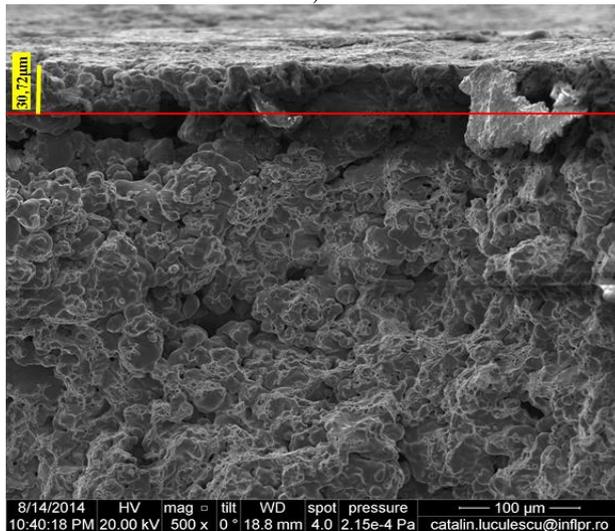
- the steels with 0,25% C and pressed at 650 MPa attained the highest values of the roughness.

Because is difficult to determine the area of densified surfaces, the samples were cut and then were studied by SEM microscopy.

In fig. 7 are presented SEM images of the sintered steels undensified and densified.



a)



b)

Fig. 7. SEM images of the steel obtained by homogenous mixture with 0,15%C pressed with 500 MPa: a) as sintered; b) densified (0,15/500-250/20)

All the layers of the sintered steels were measured and the results are presented in table 7.

Table 7. The thicknesses of the densified layers by high energy ball mill

Rotational speed [rpm]	Time [min]	Carbon content [%]			
		0,15		0,25	
		Compacting pressure [MPa]			
		500	650	500	650
Thickness of the densified layer [μm]					
150	5	20,32	18,83	10,25	9,5
	15	22,4	20,61	11,22	10,3
	20	23,81	21,5	11,91	10,52
250	5	21,02	20,65	13,28	11,24
	15	26,45	25,22	16,52	13,35
	20	30,72	28,95	18,38	15,21
350	5	28,22	26,82	21,12	18,85
	15	38,9	36,74	28,26	25,22
	20	45,91	42,25	32,92	28,72

The thickness of the densified layer is influenced by the milling parameters and by chemical composition. The layers of the densified steels after 15 respectively 20 minutes are more visible than those after 5 minutes. The thicknesses of the densified layers are in the range of [9,5-45,91] μm, the highest value of the thickness being attained for the steels with 0,15% C, pressed at 500 MPa and densified with 350 rpm for 20 minutes.

In order to determine the porosities of the densified layers were used the values mention above and by image analysis using NIS ELEMENTS software were measure the area of the pores ( $A_p$ ) and the area of the densified surfaces ( $A_{ds}$ ). The porosity was determined in accordance with the relation:

$$P = \frac{A_p}{A_{ds}} [\%] \quad (3)$$

The calculations for densities were made in accordance with relation (2).

The variation of the porosities and the densities function the densification time is presented in fig. 8.

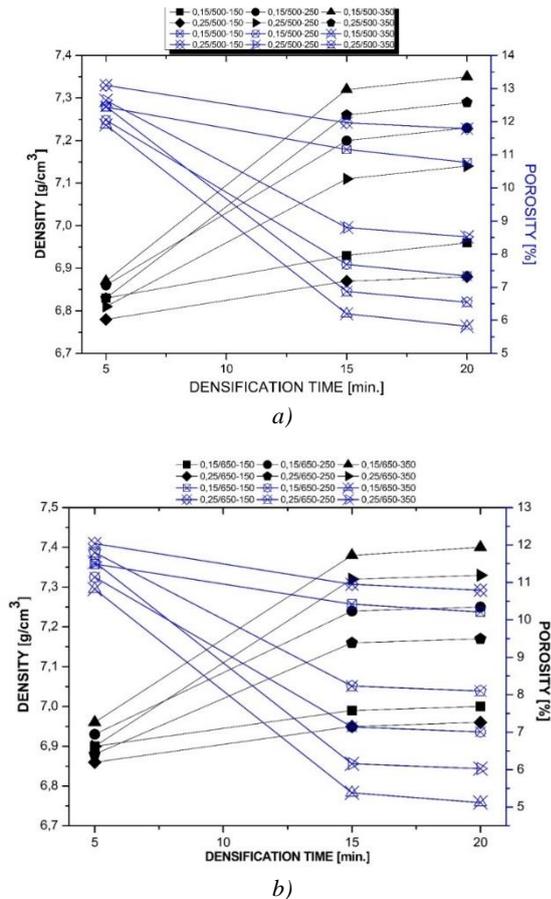


Fig. 8. Evolution of the porosity and the density: a) for the samples pressed at 500 MPa; b) for the samples pressed at 650 MPa

The sintered steels with lower C content (0,15% C) are much more sensitive for plastic deformation using ball milling comparative with those which have 0,25% C. The densification using planetary balls mill is beneficial only for rotation speeds higher than 150 rpm and densification time higher than 15 minutes. According to fig. 8 it is observed that, after 5 minutes of densification, for all the rotational speeds the density is below 7 g/cm<sup>3</sup>. After 15 minutes of densification, the density is above 7 g/cm<sup>3</sup> and it increase with the increasing of the rotational speeds. The porosity is also influenced by the densification parameters and the highest value of the porosity was attained for the sample 0,15/650-350/20. The demand of higher densities is imposed for the sintered steels prepared by PM technologies which are used for machine parts.

The charpy tests were made in accordance with standard MPIF 40 using unnotched rectangular samples, fig. 9.

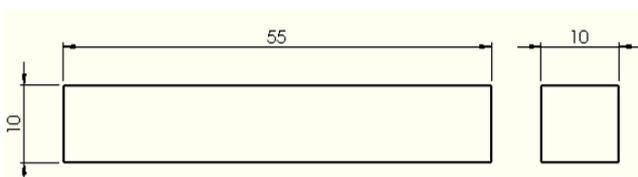


Fig. 9. Charpy test specimen

The results are plotted in fig. 10.

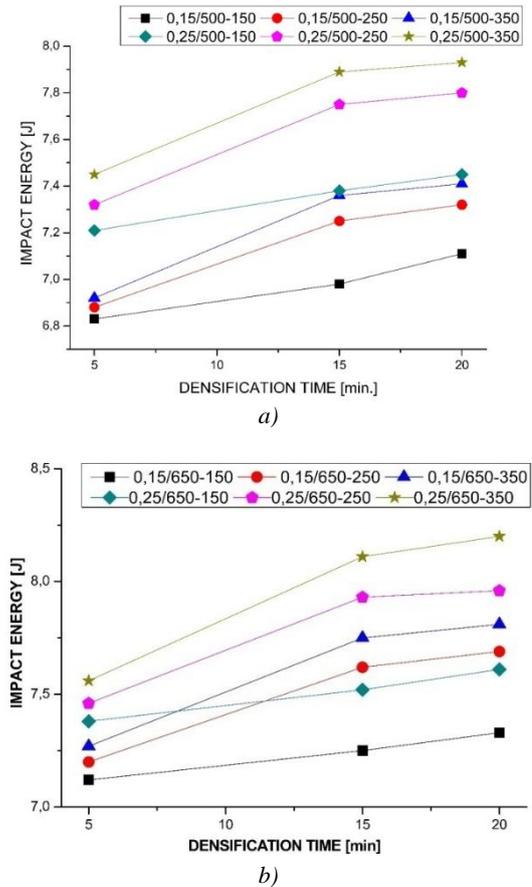


Fig. 10. Evolution of impact energy: a) for the samples pressed at 500 MPa; b) for the samples pressed at 650 MPa

In the case of sintered steels, the impact energy increases with carbon content and pressing pressure hence, the highest value was attained for the sample 0,25/650-S (7,13 J).

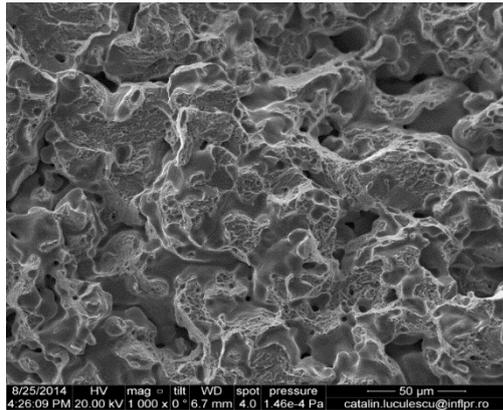
The densification process influences the impact energy of the samples. In the case of densified steels with 0,15% C the impact energy increases with (1,79-12,70)% and in the case of those with 0,25% C the increasing is about (2,41-8,20)%. The densification parameters influence the impact energy in terms of increasing the impact energy with increasing of the rotational speeds and the densification time.

Due to the lower thicknesses of the densified layers the impact energy has a lower level of increasing.

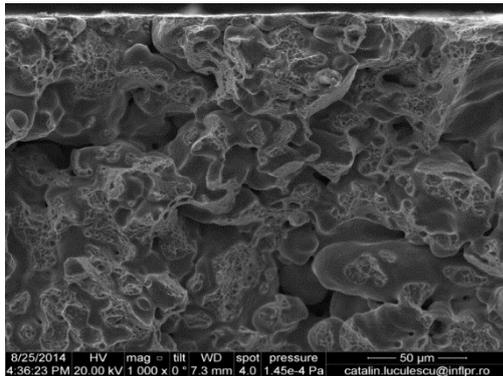
The highest value of the impact energy was attained for the sample with 0,25% C, densified at 350 rpm with a dwell time of 20 min (0,25/650-350/20).

For the steels with the densities below 7,00 g/cm<sup>3</sup> the rupture at charpy test is intergranular hence, the values of the impact energy are lower. For the steels with densities higher than 7,00 g/cm<sup>3</sup> the rupture is intragranular which assures highest values of impact energy.

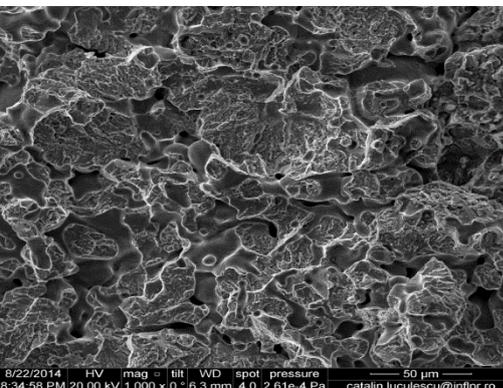
In fig. 11 is presented an example with the differences between undensified steel and the steel densified at 350 rpm with a dwell time of 20 minutes.



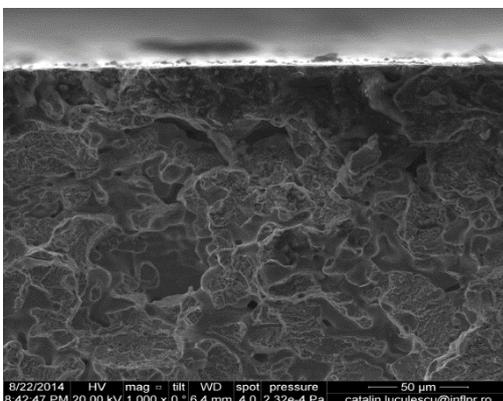
a)



b)



c)



d)

Fig. 11. SEM images: a, b) undensified steel 0,25/650-S (a - core and b - marginal layer – section view); c, d) densified steel 0,25/650-350/20 (c - core and d - marginal layer – section view)

Comparative with the sintered steel which has both the core and marginal layer with intergranular rupture in proportion of 80-85%, in the case of densified steel the rupture is intragranular in proportion of 70-75% in the marginal layer and 75-80% in the core. This shows that, by plastic deformation using a planetary ball mill, beside surface densification another densification occurs in the core but with lower intensity.

#### 4. Conclusions

Surface densification of sintered steels using a high-energy planetary ball mill is influenced by C content of steels, compacting pressure and working parameters of the planetary ball mill.

Experimental results highlight the following:

- The level of surface densification decreases with increasing of graphite content and compaction pressure of the homogeneous mixtures;

- The level of densification increases with increasing of rotational speed and dwell time at that speed;

- Comparative analysis of the superficial layers densities show that the optimal conditions are for the rotational speeds of 250 rpm with a 20 minutes dwell time;

- Densified layer thicknesses are in the micron range, the higher thickness was reached for the steel 0,15/500-350/20 namely 45.91  $\mu\text{m}$ . The thickness increases with the intensify of the milling work regime;

- Quality of the surfaces which are in contact with the grinding balls and bowl is worsens with the intensification of the milling work regime.

- The impact energy is influenced by the working process parameters and it increases with their increasing.

Based on these considerations it appears that, for the surface densification of sintered steels, cold plastic deformation using planetary ball mill can be used when it is necessary high efficiency operation and for complex configuration parts.

#### References

- [1] Francis Hanejko, High Density via Single Pressing / Single Sintering.
- [2] Lawcock, R., Buckley-Golder, K., and Sarafinchan, D., Testing of High Endurance PM Steels for Automotive Transmission Gearing Components,” SAE International Congress & Exposition, Michigan, (1999).
- [3] Strehl, R., Tooth Root Fatigue of a High Density Gear,” WZL TH Aachen, Germany.
- [4] GKN Sinter Metals, Materials and Processes.
- [5] Shoot peening applications, Metal Improvement Company, www.metalimprovement.com, (2005).
- [6] David Kirk, Shot peening, Aircraft Engineering and Aerospace Technology: An International Journal 77(4), 349 MCB University Press., ISSN 0002-2667, (1999).

- 
- [7] Marsh, K.J. (Ed.), Shot Peening- Techniques and Applications, EMAS, ISBN 0947817646, (1993).
- [8] Military Specification-Shot Peening Metal Parts, MIL-S-13165C, (1989).
- [9] Military Specification- Steel Grit, Shot and Cut Wire Shot and Iron Grit and Shot-Blast Cleaning and Peening, MIL-S-851D, (Replaced by SAE J1993, J827, J444 and J441), (1990).
- [10] Link, R. and Kotthoff, G., Suitability of High Density Powder Metal Gears for Gear Applications, Gear Technology, Jan/Feb., (2001).
- [11] Montross, C., Florea, V., Brandt, M. and Swain, M., Subsurface Properties of Laser Peened 6061-T6 Al Weldments”; Surface Engineering, (2000).
- [12] Van Aken, D., Laser Shock Processing, Industrial Heating, Feb. (2001).
- [13] Hackel, L., Harris, F., Halpin, J., Rankin, J., Hill, M. and Chen, H., The Effects of Process Variations on Residual Stress Induced by Laser Peening”; Materials Science Forum, (2002).
- [14] Lakhwinder S., R.A. Khan, M.L. Aggarwal, International Journal of Engineering Science and Technology 2(5), 818, (2010).

---

\*Corresponding author: nicolicescu\_claudiu@yahoo.com