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Aspects of the tribological behaviour of powders recycled from rapid steel treated sub-zero*

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Abstract. The recycling of high-alloyed steels represents a significant opportunity in Powder Metallurgy as it permits the use of raw materials with relatively low prices compared to the conventional methods. Recycling can be achieved by two methods: from spraying debris resulted from worn cutting tools and processes obtained from processing chip drilling and re-sharpening of tools. The research aims to confirm that wastes from rapid steels can become, by the successive processing, metal powders that can thereafter be used for cutting tools of lathe type removable plate. After pressing and sintering the recycling powder, cylindrical samples were obtained that were subsequently applied a subcritical annealing. Wear tests conducted on a tribometer type TRB-01-02541 confirmed that their wear resistance is superior to the same samples that were sintered, hardened and tempered in oil.

1. Introduction

The paper presents studies conducted by a research team from the Faculty of Mechanical Engineering of the University of Craiova on the mechanical characteristics of pieces obtained from recycled metal powders that were sintered and treated sub-zero.

Recent investigations undertaken worldwide in connection with the changes that take place in steel when subjected to very low temperature following the usual heat treatment [1, 2, 3, 4] open up an entirely new field in the treatment of cutting tools and alloyed steels in general.

Recycling of rapid steels is a significant opportunity in Powder Metallurgy because it allows the obtaining of raw materials with relatively low prices as compared to the classical powders.

In the research was used only rapid steel type HS 18-0-1 EN ISO 4957:2002, obtained by recycling steel cutting tools from same material type.

Two possibilities for achieving the recycling were identified: spraying molten metal from the remains of cutting tools and machining waste chips resulting from sharpening and re-sharpening processes of cutting tools.

Research processes aim to confirm that waste from rapid steels can be used after the successive processing by pressing, sintering and subcritical annealing of the metal powders as removable plates to knives cutting lathe. Therefore, the project benefits the environment as well as in the Powder Metallurgy industry.

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2. Experimental research

The chemical composition of the initial chips and distribution of alloying elements was determined with an electronic microscope SEM equipped with EDS spectrometer and analysis software INCA 200. The chemical composition of rapid steel chips used in research is shown in figure 1.

EDAX ZAF Quantification (Standardless)
Element Normalized
SEC Table : Default

Elem	Wt %	At %	K-Ratio	Z	A	F
C K	2.28	9.52	0.0059	1.2179	0.2131	1.0003
O K	6.20	19.41	0.0300	1.1938	0.4044	1.0024
V K	1.15	1.13	0.0120	0.9950	0.9698	1.0788
CrK	3.68	3.73	0.0432	1.0142	0.9792	1.1224
MnK	0.99	0.90	0.0097	0.9969	0.9849	1.0046
FeK	67.30	60.35	0.6796	1.0167	0.9865	1.0068
W L	18.20	4.96	0.1339	0.7430	0.9905	1.0000
Total	100.00	100.00				

Element	Net Inte.	Backgrd Inte.	Error	P/B
C K	4.72	1.82	4.33	2.60
O K	53.48	2.44	1.01	21.87
V K	7.51	7.45	4.46	1.01
CrK	22.69	6.98	1.89	3.25
MnK	4.22	6.05	6.77	0.70
FeK	239.10	5.55	0.47	43.08
W L	6.82	3.59	3.88	1.90

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kV: 15.01 Tilt: 0.00 Take-off: 35.80 AmpT: 102.4
Det Type:SUTW, Sapphire Res: 130.42 Lsec: 200

Figure 1. Parameters and results of EDAX analysis.

Original chips are generally rough and irregular shaped needle containing abrasive grains on the surface, therefore magnetic separation is required. After 10 hours of grinding the grain powder is in the range [0.1 to 150] μm and after 20 hours of grinding grain powder is in the range [0.04 to 25] μm.

Both the chemical analysis of the chips used in research as well as the working procedure used for obtaining recycled powder was described in detail in the paper [5].

Recycled powder from rapid steel was compacted by into an electro-hydraulic press and then unilaterally pressed into a mold of Ø12 mm at a pressure of 800 MPa, as shown in figure 2. Subsequently, the sintering process of green samples was carried in a sealed box in a neutral atmosphere (Argon) at a rate of 2.5 l/h at three different temperatures: 1150°C, 1200°C and 1250°C.

The research conducted used a constant rate of heating in order to ensure a uniform distribution of the heat flow in the total mass of parts, thereby reducing the possibility of cracks or internal defects occurring. The best density values for the sintered parts were obtained when two combined conditions were used: maximum compacting pressure and the grain aggregate as small as possible [6]. One of the main objectives of the study was to obtain the compacts with good wear resistance, which can then be used as removable plates in the cutting process.

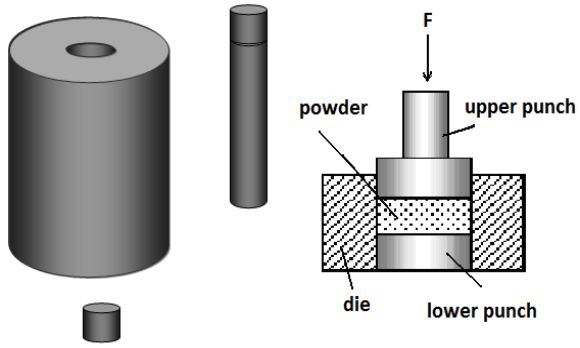
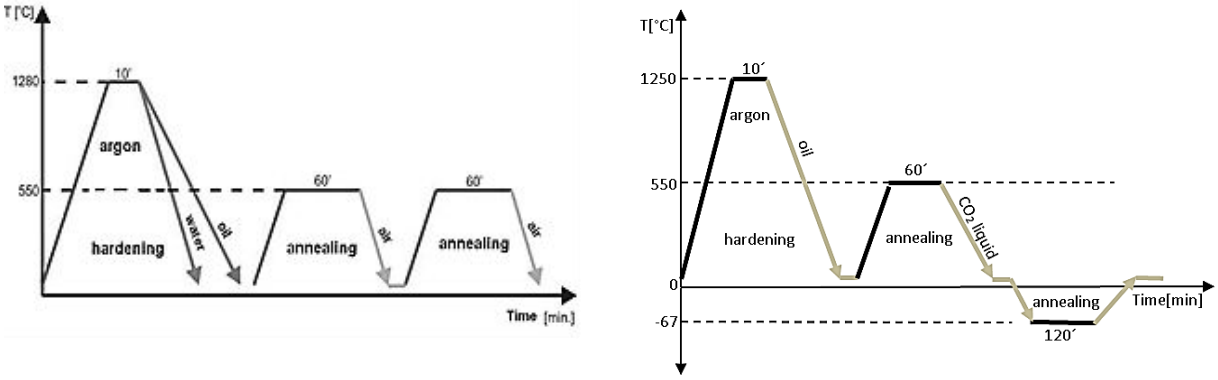


Figure 2. Unidirectional cylindrical mold.

According to the measurements conducted in the study, the density of samples sintered at 1150 °C samples ranged between [4.56 to 6.14] g/cm³, at the samples sintered at 1200 °C ranged between [4.89 to 6.38] g/cm ³, and at the samples sintered at 1250°C, the density of compacted samples was in the range [5.21to 6.75] g/cm³. The porosity of the compacts is complementary to the density, the lowest porosity of 13.46% was recorded for sintered samples at 1250°C. Also after sintering, the samples were cooled in the furnace under with protective atmosphere of argon throughout process.

In order to ensure a high wear resistance of the samples of rapid steels recovered powder, compacted at a pressure of 800 MPa and sintered under the conditions mentioned above, the samples were thereafter subjected to two types of heat treatment:

- hardening heat treatment and oil annealing, as shown in diagram of figure 3a
- hardening treatment sub-zero, according to the diagram in figure 3b

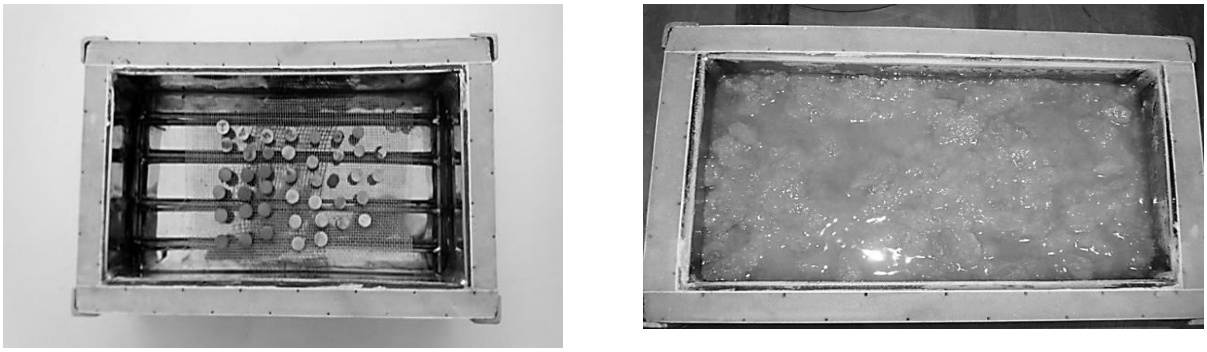


(a) (b)

Figure 3. Diagram of hardening and annealing: oil (a) and the diagram of hardening and annealing sub-zero (b).

Before adding samples in dry ice, they were treated by oil annealing. The procedure applied for the sub-zero treatment was as following: [7, 8].

- sub-zero thermal treatment was carried out in a mixture of dry ice (solidified CO_2) and technical alcohol, according figure 3.
- in order to maintain the proposed 206 K temperature, dry ice was added during the long sub-zero treatment (120 min);
- treatment dry ice was carried out in a thermally insulated box.



(a) (b)

Figure 4. Details on how to achieve the sub-zero treatment: (a) box insulation used for cooling samples (b) the thermal treatment with dry ice.

Dry ice thermal treatment parameters are shown in table 1.

Table 1. Parameters of treatment dry ice.

Treatment	The procedure	Parameters
Treatment with dry ice	The temperature of the cooling	206K (-67°C)
	Cooling medium	solution CO_2 (dry ice)
	Retention time	τ_1 = 120 min
	The rate of cooling	25^o C/min

3. Results and discussion

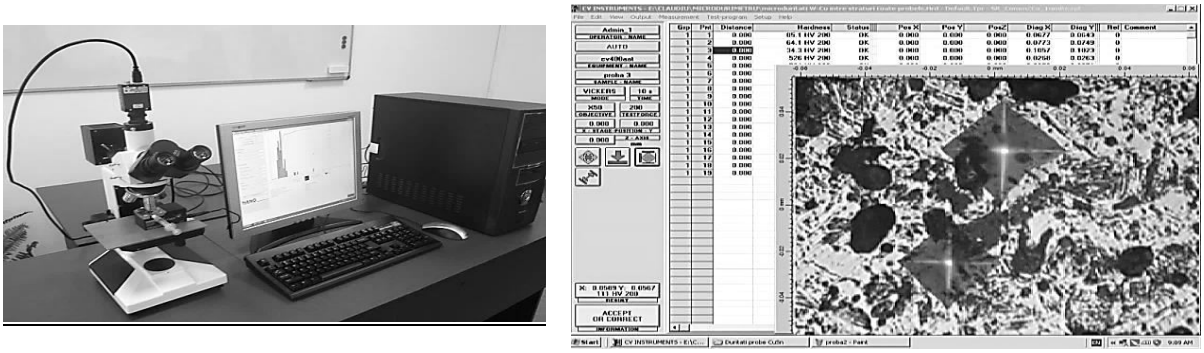
Heat treatments have led to the transformation of residual austenite into martensite and of partial decomposition of martensite and carbides in finely dispersed precipitation [9, 10].

The sintered parts were embedded in epoxy resin using an embedded machine brand METAPRESS-A after which they were prepared metallographic on a grinding machine type FORCIPOL with two workstations and speed platters between 50 and 500 rpm.

Two types of measurements were performed on the annealed samples. In the first stage the hardness of the samples was determined after heat treatment and oil annealing and in the second stage the hardness was determined after thermal treatment and annealing sub-zero in ice dry, followed by the measuring the friction coefficients in both situations After polishing the surface of the samples, it was attacked with alcohol and NITAL solution in concentrate 2%

Hardness measurements were performed with Vickers Micro-Hardness System CV-400DM^{TM}, from CV INSTRUMENTS EUROPE BV, according to figure 4.

The hardness after the application of the two variants of heat treatment is shown in figure 5.



(a) (b)

Figure 5. Hardness measuring equipment:
(a) Vickers Micro-Hardness System CV-400DM^{TM} and
(b) hardness measurement mark.

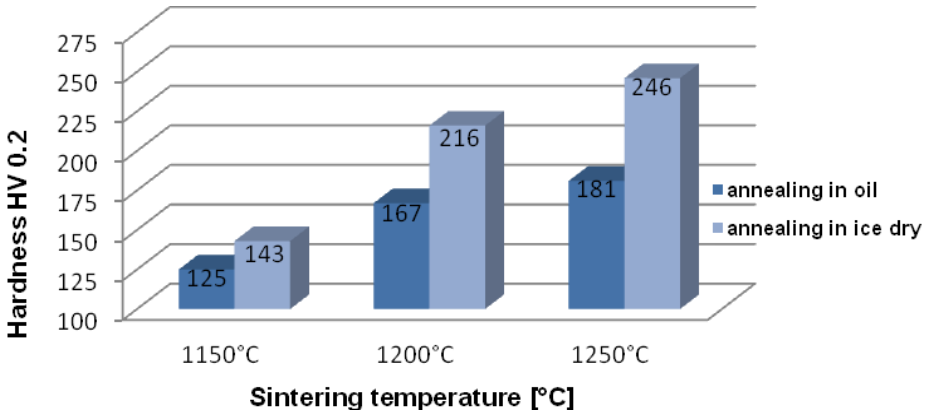
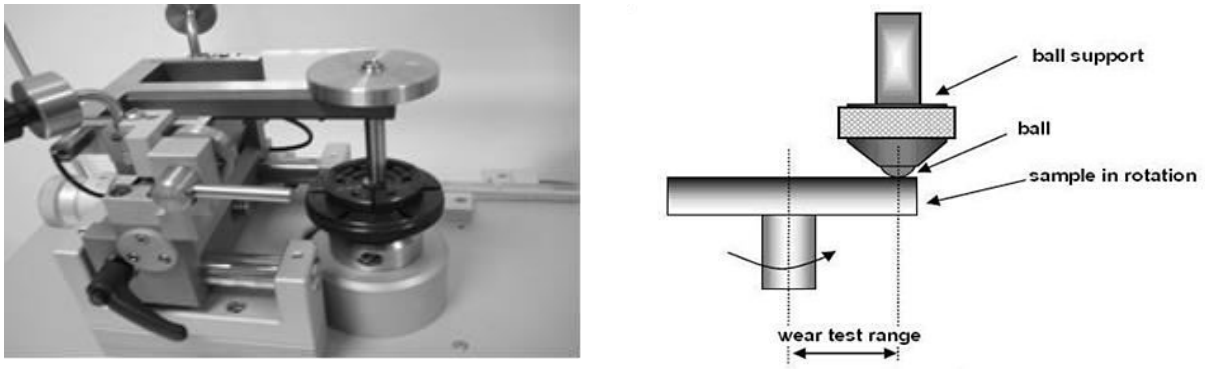


Figure 6. The hardness values according to applied heat treatment.

For evaluating the friction coefficient a tribometer brand CSM Instruments, type TRN 01-02541 was used, which allowed for the determination of friction under pure sliding friction torque using Class I under dry friction. The way of measuring the friction coefficient is shown in figure 7.



(a) (b)

Figure 7. Measuring of cylindrical parts wear coefficient:
(a) tribometer type TRN 01-02541 and (b) the measurement principle.

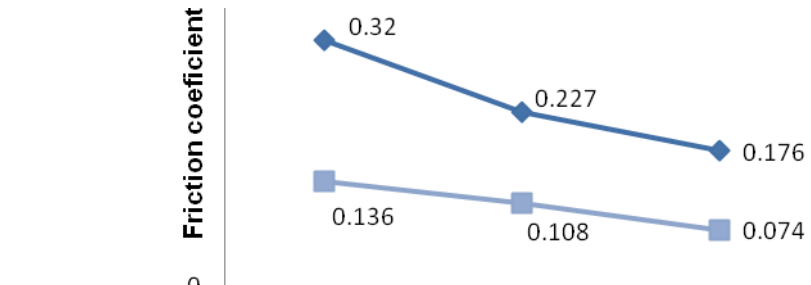
The test parameters used to determine the coefficient of friction were:

- working version: linear sliding;
- normal force: 2 N;
- radius: 3 mm;
- sliding speed: 1 cm/s;
- 100Cr6 steel ball, diameter 6 mm;
- working environment: air;
- air temperature: 27.3 ° C;
- relative humidity RH = 35.5%.

The tribological behaviour of sintered samples was tested in two different situations, and the average values of friction coefficients it is shown in figure 7:

- after sintering treatment followed by oil annealing;
- after sintering, treatment followed by annealing sub-zero.

The data on the tribological behaviour of sintered compacts from powder rapid steel recovered, results in the experimental research, revealed that the friction coefficient depends on the compaction pressure, the parameters of the process of sintering (temperature and dwell time) and the applied thermal treatment [11].



	1150°C	1200°C	1250°C
annealing in oil	0.32	0.227	0.176
annealing in ice dry	0.136	0.108	0.074

Figure 8. The evolution of friction coefficient depending on thermal treatment.

4. Conclusions

Research conducted on samples of rapid steel sintered and hardened in advance and then annealed at sub-zero temperatures shows structural improvements compared to sintered steels hardened in oil, so in conclusion it was observed that they had a high resistance to wear which means an increase in life cycle.

The role of sub-zero treatment is to make it possible to reduce residual austenite in the steel and martensite transformation to obtaining an improved hardness, good wear resistance and dimensional stability.

After applying thermal treatment sub-zero, it was observed an increase by approximately 30% in hardness, comparative to conventional thermal treatment applied of rapid steel and low friction coefficients and thus a greater durability that can justify the application in practice of this thermal treatment even if the result will increase final costs.

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