ADVANCES
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ADEM 2012

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The Organising Committee whishes to all participants at ADEM 2012 "Welcome at the Department of Engineering and Management of Technological Systems, Faculty of Mechanics, University of Craiova!"

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Foreword

The International Conference ***ADvances in Engineering and Management*** - ***ADEM 2012*** organized by the Department of Engineering and Management of Technological Systems, Faculty of Mechanics, University of Craiova has reached the second edition. The first one was held in 2010 organized by Faculty of Engineering and Management of Technological Systems Drobeta Turnu-Severin, University of Craiova and represented a significant scientific event with a broad audience both national and international.

The ADEM 2012 International Conference attracted more than 50 participants from 4 countries, representing prestigious universities, research institutes and industry. The Conference Proceedings contains 49 full papers that have been submitted for this scientific event.

The present volume provides up-to-date, comprehensive and worldwide state-of-the art knowledge in Advanced Materials & Engineering, including:

- Advanced Materials and Technologies (18 papers);
- Environment Engineering for Sustainable development (15 papers);
- Modeling and simulation (9 papers);
- Inland navigation and logistics (7 papers);

The authors and co-authors are solely responsible for the intellectual content of the papers.

As a chairman of the Organizing Committee of ADEM 2012, I would like to thank to all the authors and co-authors of the papers and to the members of the Scientific Committee for their efforts in reviewing the scientific papers. My special thanks are due to the invited speakers and to the authors from abroad for attending the conference and for their willingness to share knowledge from their research and academic experience. The organizers are thankful to the generous sponsors for their financial support of this scientific event

Drobeta Turnu-Severin, December 2012

Professor Gabriel Benga, PhD
Chairman of the Organizing Committee of ADEM 2012

RESEARCH AND DEVELOPMENT AT IMST DEPARTMENT

Gabriel Benga

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Abstract. *The paper presents a wide spectrum of the range of engineering activities starting from the nanostructures of the materials and ending at advanced technologies. The variety of the themes presented offer the opportunity to get an inside view on the research activities performed at Department of Engineering and Management of Technological Systems*

Keywords: *sintered materials, magnetic materials, biocomposite materials, microwave sintering, microwelding*

1. INTRODUCTION

Engineering research is one of the main sources of innovative ideas and their consequences, advances in materials and technologies play a major role in our lives and our future being in this way of the utmost importance.

The wide end rich palette of the introduced results covers lots of segments of Materials Science and Industrial Engineering from the conceptual design through technology and planning, ranging from control and management to experiments. From high performance, economical and environmental point of view, Powder Metallurgy (PM) shows remarkable advantages in production of parts and components due to their special composition by elemental mixing and three dimensional near net shape forming methods. PM process can be applied not only to metal materials but also ceramics and organic materials.

Composite materials, often shortened to composites are engineered or naturally occurring materials made from two or more constituent materials with significantly different physical or chemical properties which remain separate at the macroscopic or microscopic scale within the finished structure.

2. RESEARCH ON THE ELABORATION OF WEAR RESISTANT SINTERED STEEL BY GAS-CARBU-SINTERING (GCS) PROCESS

PM steels, made of Fe+0,1% graphite powder mixture, have been processed by carburizing + sintering operations in one single treatment step. The wear behavior is studied from the point of view of the technological parameters: carburizing and sintering times and temperatures. [1]

Sintered steels are widely used in fabrication process of different parts in power tools industry, automotive, agriculture, construction. Wear behavior of sintered steels may focus on wear transitions in sliding contacts because majority of machine parts have sliding movements. [2] The morphological properties of the Fe powder particles are presented in fig. 1. The Fe powders (DWP 200) are from S.C. Ductil Buzau, Romania.

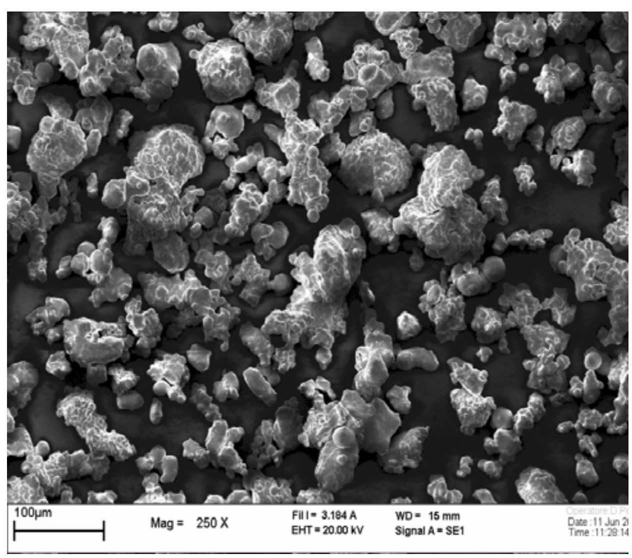


Fig.1. Morphological aspects of Fe (DWP200) powder particles

Two types of PM steels have been processed, as it is presented in Tab.1, 2 and fig. 2.

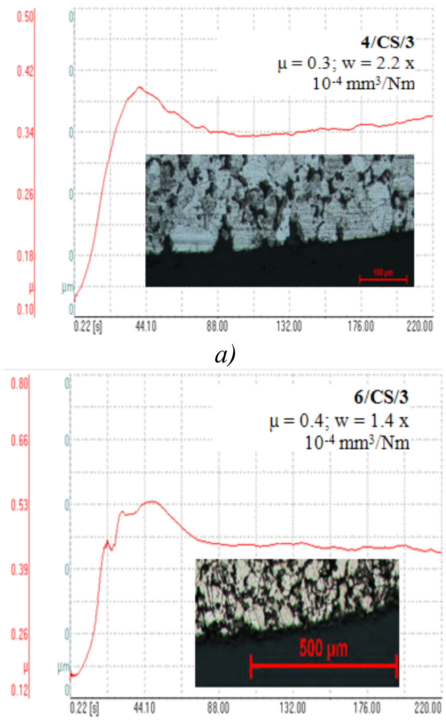
Tab. 1. Processing technologies for PM steels

Heat treatment / Sample code	CONVENTIONAL ROUTE: SINTERING + CARBURISING (S+C)					
	step 1			step 2		
	SINTERING			CARBURISING		
	gas atmosphere	treatment temp. [T^0C]	soaking time τ [min.]	gas atmosphere	treatment temp. [T^0C]	soaking time τ [min.]
S+C	argon	1150	60	CH_4	910	30 60

Heat treatment / Sample code	New route: CARBU-SINT (CS)					
	one single step					
	CARBURISING			SINTERING		
	gas atmosphere	treatment temp. [T0C]	soaking time τ [min.]	gas atmosphere	treatment temp. [T^0C]	soaking time τ [min.]
CS	CH4	910	30 60	argon	1150	60

Tab. 2. Sintered PM steels for dry friction and wear tests

Sample code	Processing technology by heat treatment	Wear tests conditions
C+S	step1: sintering step 2: carburising	Equipments: ➢ tribometer TRB 01-02541 (CSM Instruments S.A. Elvetia), soft InstrumX version 2.5A for friction coefficient measurement, μ ➢ Surtronic profilometer 25 (M112/3522-01), Taylor Hobson Precision (U.K.), software TalyProfile Silver for wear section area [μm^2] and wearrate [mm^3/Nm] measurement friction couple: ball (counterpiece) on disc sample rotation speed: 1 cm/s normal load of the ball on disc: 9 N ball: 100Cr6; diameter 6 mm; Ra < 3,2 μm; HRc 60-64; density > 7,6 g/cm^3 friction conditions: dry testing atmosphere: air, 25^{0}C atmosphere humidity: 30%



a)

b)

Fig. 2. Variation of the friction coefficient for the CS steels along the dry friction tests: a) 4/CS/3; b) 6/CS/3

The detailed analysis of the wear behavior of the PM steels processed by CARBUSINT and conventional PM routes allows the following conclusions to be stated:

- about the processing technology, the CARBUSINT route is superior to the conventional one, considering the wear behavior of the processed steels as well as the technical-economical advantages,
- the low carbon content (0,1% C as graphite flakes in the powder mixture) provides a good wear strength. The CARBUSINT steels present different wear rates on the surface (w = 1,4 x 10^{-5} mm^3/Nm) which is a very good value in comparison with the core area (w = 3,9 ... 4,3 x 10^{-4} mm^3/Nm),
- on the compaction pressure, the higher it is the better the wear behavior for the CARBUSINT steels. As far as concern the conventional steels, the compaction pressure has no influence on the wear rate,
- the carburising soaking time plays an important role for the CARBUSINT steels. For 60 min. carburizing soaking time, specific structural components are synthesized, providing high wear strength on the steels surface.

The present study underlines that the CARBUSINT technology is recommended to obtain PM steels with high wear rate on the top surface of the products and high toughness in the core region. [1]

3. RESEARCH ON SOLID STATE CARBURIZING OF SINTERED STEELS FOR AUTOMOTIVE PARTS

The carburizing treatment is important for the sintered steel parts especially for those products with complex loadings for the automotive industry. For the sintered steels, the carburizing specific processes are different because of the structural porosity. Thus, in case of powder metallurgy, the carburizing provides not only the enrichment of the superficial layer in C% but also in the bulk part in order to elaborate functionally graded sintered steels considering the concentration and properties. This research is focused on the solid state carburizing of sintered steels concerning their porosity and C % content. [3] Also, the approaching of the carburizing treatment from green compact state – CARBSINT – as well as the carburizing from sintered state – SINTCARB – is discussed.

Iron powder particles type DWP 200 – HEG5001 mixed with 0,15% respectively 0,25% graphite powders and 1% Zn stearate have been used. The mixtures have been homogenized for 30 minutes in TURBULA homogenizer and then cold compacted at 500 respectively 650 MPa. The samples are cuboids of 10x10x55 mm corresponding to ASTM E23, ISO 5754 for the toughness testing. The green compacts have been thermal processed by SINTCARB following the thermal cycles presented in fig. 3 respectively by CARBSINT as it is shown in fig. 4. The solid state carburizing was carried on using the carburizing slurry made of 85% smut, 10% Na_2CO_3 and 5% fuel oil. The carburizing slurry was thermal analyzed by DTA and the carburizing was determined to be in the range of 850-1150^{0}C. The carburizing time was 90 minutes for each process in order to reach the C% content gradient across each sample section. After the carburizing by SINTCARB and CARBSINT, the samples were quenched in water respectively oil and then tested for toughness determination.

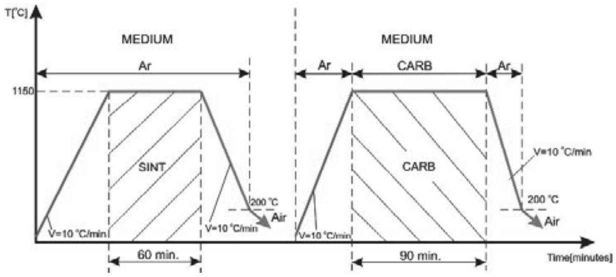


Fig. 3. SINTCARB thermal cycle

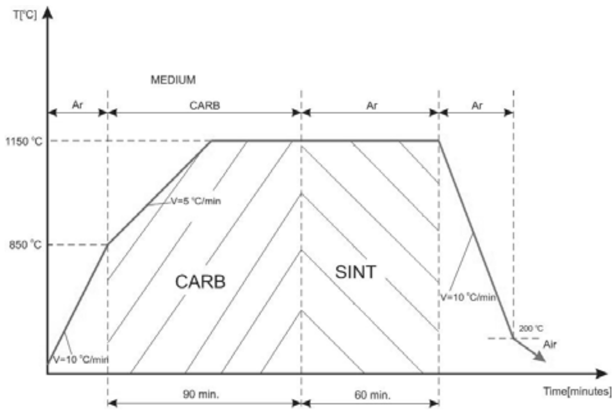


Fig. 4. SINTCARB thermal cycle

After every processing cycle, the samples have been analyzed from the point of view of density, porosity, optical microstructures and C% enriched layers. According to SRISO 14284:2003, samples of flakes were removed from the processed steels, from surface and core areas, in order to determine the C% content. The experimental results are presented in tab. 3 and the microstructural features, at three stages of the treatments, are presented in fig. 5.

Tab. 3. Physical characteristics of the samples

PARAMETERS \ SAMPLE		15-50				15-65				25-50				25-65			
		D	S	CS	SC	D	S	CS	SC	D	S	CS	SC	D	S	CS	SC
APPARENT DENSITY [g/cm³]		6,85	6,93	7,12	7,19	6,9	7,04	7,3	7,39	6,75	6,79	7,29	7,34	6,84	6,86	7,13	7,3
POROSITY [%]		12,15	11,19	8,75	7,88	11,52	9,74	6,46	5,26	13,41	12,95	6,54	5,88	12,35	12,05	8,59	6,42
Layer thickness x10³[µm]				1,39	0,98			1,15	0,92			1,35	0,98			0,68	0,33
%C	Layer	-	0,05	0,73	0,69	-	0,12	0,75	0,67	-	0,19	0,72	0,71	-	0,21	0,65	0,62
	Core			0,62	0,31			0,59	0,19			0,43	0,37			0,42	0,29
KC [J]	Not-quenched	-	20,5	11,7	12,3	-	23	15,7	14,7	-	21,7	15	12,7	-	23,5	17,7	16,7
	Water quenching	-	20	4	5,7	-	10	3	5,3	-	18	6	7	-	20	5	6,7
	Oil quenching	-	16	6	7,5	-	17	5	6,7	-	20	11	8,7	-	19	14	5,3

***D= de-binded green part, S=sintered; CS=CARBSINT; SC=SINTCARB

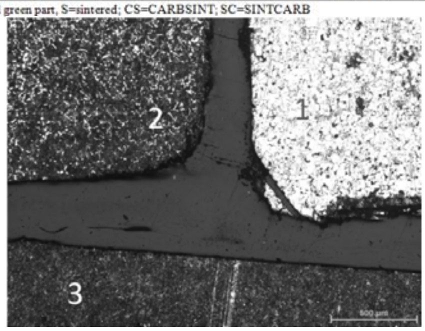


Fig. 5. Samples microstructures: 1=15-50S; 2=15-50SC; 3=15-50CS (75X)

The following outlines can be stated after the analysis of the experimental results:

- Using different carburizing treatments, sintered steels with thick carburized layers respectively %C gradient in cross section can be obtained;
- The carburized layers by CARBSINT are thicker than those processed by the conventional SINTCARB. Thus, considering the four steels type, the thickness of the carburized layers belong to (1155,35-1397,55) µm for CARBSINT respectively (331,43-985,41) µm for the SINTCARB treatment;
- The C% content is variable in the steels cross-section after the carburizing and for the core case it doesn't correspond to the graphite content in the initial mixture respectively to the diffused C during the sintering. The differences between the C% content in surface vs. the core for the carburized steels are about (0,11-0,16) % C for the steels processed by CARBSINT from homogeneous mixture with 0,15% graphite respectively (0,23-0,29)%C for the steels processed with 0,25% graphite. The same steels, but processed by SINTCARB, present (0,32-0,48)%C respectively (0,29-0,33)%C as differences between the C% in surface vs. the core.
- The thickness of the C% enriched layer and the C% in the steels cross section are related parameters and they are influenced by the porosity and the initial state of the samples before the carburizing. Thus, the thickest C% enriched layers respectively C% in the core case correspond to the initial porosity (11,52-13,41)% for the steels processed by CARBSINT vs. the sintered and carburized steels that, after the sintering stage, presented the porosity for (11,19-12,95)%;
- The toughness test results for the carburized and water quenched samples confirm the experimental results concerning the C% gradient content in the cross section. Thus, the CARBSINT samples registered the toughness values of (3-6) J after the water quenching and the SINTCARB samples have (5,3-7) J. The lower toughness values of the carburized and sintered steels are due to the large bulk C% content that corresponds to the large martensite content after the quenching.
- The evolution of the bulk C% content for the steels processed from Fe + graphite powder mixtures related to the porosity and thermal treatment used for the solid state carburizing by the means of a carburizing slurry represent the aim of this research.
- Two carburizing methods have been approached, namely the carburizing of the green compacts – CARBSINT respectively the carburizing after the sintering (SINTCARB).
- For the CARBSINT method, the C% content enrichment has been registered, the core of the samples having higher C% content of 0,4% C than the SINTCARB samples that registered (0,19-0,37)% C. As far as concern the outer layers, the C content was about 0,65%, no matter the carburizing method was used. The variation of the concentrations must be related to the C% content diffused in Fe during the

sintering stage, namely (0,05-0,12)%C for the steels made of mixtures containing 0,15% graphite respectively (0,19-0,21)%C for the steels made of mixtures containing 0,25% graphite.

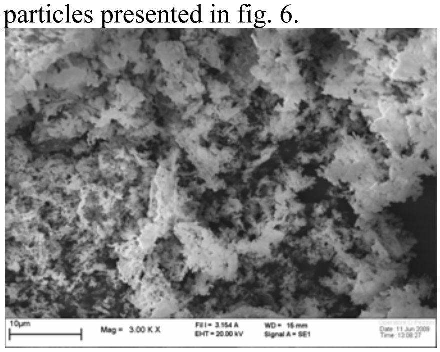
4. MAGNETIC MATERIALS BASED ON BARIUM HEXAFERRITE

$BaCO_3$ and α-Fe_2O_3 are the initial powders which were used in research to develop barium hexaferrite. The powders were milled in a high energy ball milling machine Pulverisette 4.

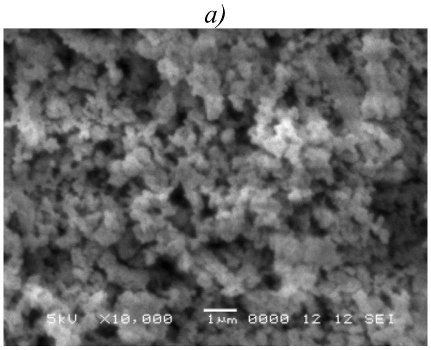
Barium hexaferrite is obtained by heating the stoichiometric homogeneous mixture of $BaCO_3$ and α – Fe_2O_3 initial powder according to the reaction below:

$$BaCO_3 + 6Fe_2O_3 \rightarrow BaFe_{12}O_{19} + CO_2$$

Some samples milled in wet medium were taken from the mill after 5 and 20 hours of milling and were analyzed by SEM to determine the shape and size of powder particles presented in fig. 6.



a)



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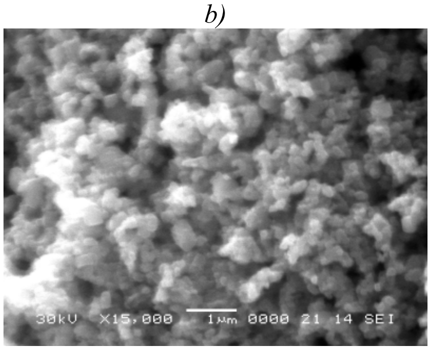


c)

Fig. 6. SEM images for mixed $BaCO_3x6\alpha$-Fe_2O_3 powders: a) homogeneous stoichiometric mixture; b) mechanically alloyed mixture for 5 hours; c) mechanically alloyed mixture for 20 hours

The microscopic aspects reveal the presence of polygonal-shaped grains for both samples mechanically alloyed for 5 and 20 hours. Small particles can be observed in the SEM micrograph for the 20 h of mechanical alloyed mixture. [4]

The heating of $BaCO_3x6\alpha$-Fe_2O_3 mixture resulting from mechanical alloying for 5 hours was carried out using a MOM Budapest derivatograph and is presented in fig. 7.

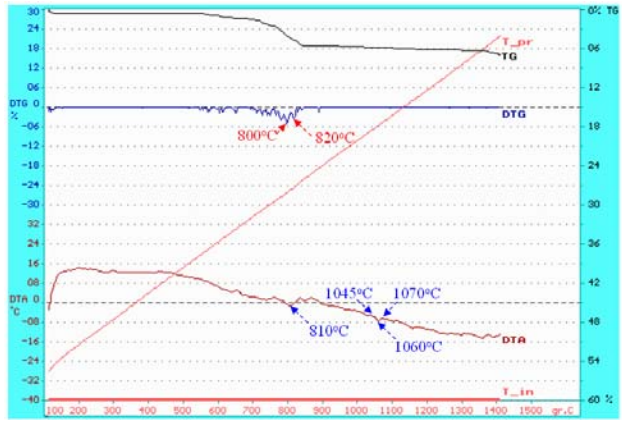


Fig. 7. The diagram of mechanically alloyed mixture for 5 hours, MA5h

In this diagram, we identify the endothermic peak from 810°C corresponding to monoferrite formation and another peak at 1060°C corresponding to barium hexaferrite formation according to the X-ray diffraction. Total weight loss at heating process of this powder is 7 %. In tab. 4 are presented the results of phase transformations at different mechanical alloying times.

Tab. 4 Values of phase transformations at barium hexaferrite formation

Sample No.	Sample code	Values of phase transformations for monoferrite (°C)	Values of phase transformations for hexaferrite (°C)			ΔT	
			Start reaction	Maximum reaction	End reaction	°C	[%]
1	HM	**850**	**1090**	**1100**	**1118**	**0**	**0**
2	MA5h	810	1045	1060	1070	40	3,64
3	MA20h	795	965	980	995	120	10,91

Compared with the reference mixture, HM, it can be observed a decrease of the formation temperature up to 10,91 % in the case of mechanically alloyed mixture for 20 hours. Therefore, it can be seen that the temperature of barium hexaferrite formation is reduced with increasing of the mechanical alloying time, namely, once with decreasing of powders particle size.

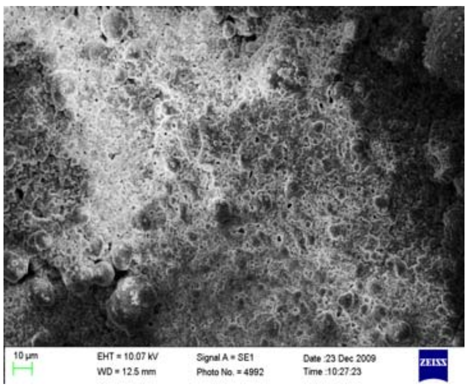
As a general conclusion, the results of experimental investigations from this research recommends for ferritisation the mixture obtained from $BaCO_3x6\alpha$-Fe_2O_3 after 20 hour of mechanical alloying in order to obtain ferrites with high performance in optimal heating conditions and high energy efficiency.

5. BIOCOMPOSITE MATERIALS

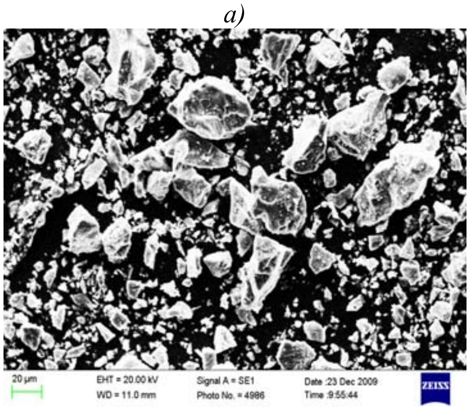
5.1. Biocomposite material elaboration

Bone grafting is a usual technique commonly applied in order to repair the hard tissue. From the point of view of the materials dedicated to this purpose, the biomaterials (metallic, ceramics or their composites) are used for grafts processing.

In the present research work HAP nanometric powder particles (fig. 8 a) have been reinforced with micronic Ti powders (fig. 8 b) in order to study the wear behaviour of these PM biocomposites during dry friction tests. These materials were chosen mainly due to their biocompatibility and their wide applications in the biomedical field.



a)



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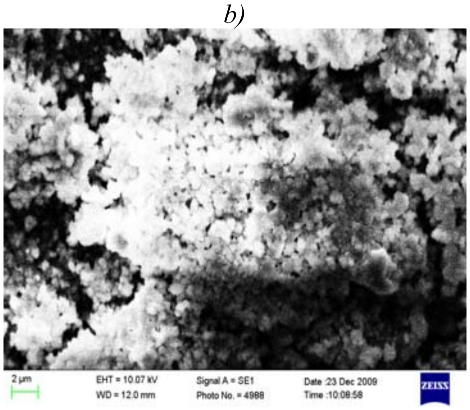

c)

Fig. 8. SEM images: a) hydroxyapatite powder; b) titanium powder; c) Hap/Ti biocomposite material

The flexibility in choosing the reinforcing ratio as well as the characteristics of the components (matrix and reinforcement) is provided by PM technology.

This processing route, also allows selecting the powder particles size, shape, chemical composition (elemental or pre-alloyed particles) that has a great influence on the biocomposite properties. In the same time, anisotropic biocomposites can be processed by PM technology, due to the modern routes tailoring the porosity/density, hardness, and mechanical properties.

SPS and TSS are ones of the PM advanced sintering techniques allowing nanostructured materials processing. The biocomposite samples were sintered using spark plasma sintering (SPS) and two step sintering (TSS) routes, fig. 9. Shorter sintering times and lower sintering temperatures represent the great advantages of SPS route.

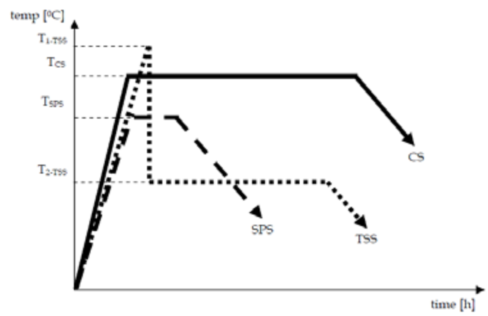

Fig. 9. Schematically representation of thermal cycles in the case of classic sintering (CS), spark plasma sintering (SPS) and two steps sintering (TSS)

The wear tests have been performed on the TRB 01-02541tribometer (CSM Instruments SA, Switzerland), with the linear reciprocating module, equipped with InstrumX software, version 2.5A. The main characteristics of the tribometer are:

- maximum torque =450Nmm;
- maximum load = 46 N;
- frequency up to 1.6 Hz;
- linear speed range = (0.3–500) mm/s;
- stroke range = 60mm;

The wear rates as well as the profile of the wear track are determined by using the Surtronic 25 profilometer (M112/3522-01), from Taylor Hobson Precision (England), equipped with TalyProfile Silver software for the technical determinations.

In this research work several possibilities to obtain nanostructured HAP/TI based biocomposites have been presented pointing out especially the TSS and SPS techniques. [5]

5.2. Laser micromachining of HAP based biocomposites

The micromachining of the HAP/Ti biocomposites was performed on a LASAG KLS 246 pulsed Nd:YAG laser for industrial materials processing. The laser parameters: voltage [V], pulse frequency [Hz] and pulse