The Influence of Barium Carbonate on the Tribological Characteristics of Some Iron-based Friction Composites

MERIE Violeta¹, a *, CÂNDEA Viorel², b and POPA Cătălin³,c

¹Technical University of Cluj-Napoca, Faculty of Machines Building, Department of Mechanical Systems Engineering, 103-105 Muncii Avenue, 400641, Cluj-Napoca, Romania
², ³Technical University of Cluj-Napoca, Faculty of Materials and Environmental Engineering, Department of Materials Science and Engineering, 103-105 Muncii Avenue, 400641, Cluj-Napoca, Romania

a *Violeta.Merie@stm.utcluj.ro, b Viorel.Candea@stm.utcluj.ro, c Catalin.Popa@stm.utcluj.ro

Keywords: Iron-based friction composites, powder metallurgy, barium carbonate, tribological behavior, microstructure.

Abstract. New iron-based composite materials with addition of barium carbonate (2 to 8 wt% barium carbonate) for friction applications are investigated. The tribological behavior of the studied materials was determined by a pin-on-disk method when a cast iron disk was employed. The addition of 2 wt% barium carbonate determined a significant increase of the average friction coefficient. Instead a further increase of barium carbonate content determined a gradual decrease of this parameter. The improvement of wear resistance was marked out for a barium carbonate content of up to 6 wt%. The optimal ratio between the average friction coefficient and the wear rate for the researched Fe-Cu-graphite-Ni-BaCO₃ composites was determined for the iron-based material containing 2 wt% barium carbonate. SEM, EDX and XRD analysis marked out a complex structure containing alloyed ferrite, pearlite and carbides, traces of nickel and barium carbonate and free graphite.

Introduction

Friction materials are materials that have to fulfill some requirements such as high friction coefficient, good wear resistance, good thermal conductivity and corrosion resistance, long life in service etc. [1, 2]. All the properties mentioned above have to be stable in time, at high temperatures and high charging loads. Iron-based friction composites are complex materials containing an iron or iron alloy matrix and other metallic, nonmetallic and/or organic materials which have to increase the friction coefficient and to improve the wear resistance [3]. The high friction coefficient and mechanical characteristics as well as the good wear resistance and the economy of raw materials are some aspects that recommend the usage of iron-based friction composites for manufacturing friction products. Due to their complex compositions, the friction composites are intensively investigated in order to establish the composition-structure-properties relation [4, 5]. The applications of these materials are the everyday ones (shoes sole), in automotive (braking system) and biomedical (knee prosthesis) industries, the fabrication of some components for office equipment or house equipment and so on [6].

The influence of the solid lubricants, the influence of friction components, the influence of the content of the components that have to strengthen the metallic matrix as well as the influence of technological parameters on the tribological characteristics of iron-based composites have been extensively studied [7-13].

This study is a research concerning the influence of barium carbonate content on the tribological and structural characteristics of a Fe-10%Cu-7%graphite-12%Ni friction composite obtained by a powder metallurgy method.
Materials and experimental procedure

Materials

Four iron-based friction composites were investigated in order to highlight the influence of barium carbonate content on the tribological characteristics of a Fe-Cu-graphite-Ni material. The chemical composition of the studied materials is given in Table 1. The copper, graphite and nickel contents were kept constant at 10, 7 and 12 wt% respectively while the barium carbonate content was ranged between 2 and 8 wt%.

| Material | Chemical composition (wt%) |
|----------|----------------------------|
|          | Iron | Copper | Graphite | Nickel | Barium carbonate |
| FCBC 69  | 69   | 10     | 7        | 12     | 2                |
| FCBC 67  | 67   | 10     | 7        | 12     | 4                |
| FCBC 65  | 65   | 10     | 7        | 12     | 6                |
| FCBC 63  | 63   | 10     | 7        | 12     | 8                |

The raw materials (iron, copper, graphite, nickel and barium carbonate) were added in a powder state. Some characteristics of the initial powders are given in Table 2. Iron and copper powders with particles size smaller than 100 µm were employed while the particles size of graphite, nickel and barium carbonate powders was smaller than 10 µm. A good compressibility of the studied mixtures was obtained by using powders that contain particles of different shapes.

| Powder          | Apparent density (g/cm³) | Particles size (µm) | Particles shape  |
|-----------------|--------------------------|---------------------|------------------|
| Iron FC 100.24  | 2.60                     | < 100               | Irregular        |
| Cooper          | 2.42                     | < 100               | Spherical        |
| Graphite        | 0.13                     | < 10                | Flakes           |
| Nickel          | 4.31                     | < 10                | Spherical        |
| Barium carbonate| 0.87                     | < 10                | Tabular          |

Experimental procedure

The researched composites were obtained via Powder Metallurgy route. After the mixtures were weighted according to the mixing formulas from Table 1, a mechanical milling in a Fritsch Pulverisette 4 planetary balls mill was done in order to assure a good homogeneity and a good distribution of the initial powders in the mixtures. The milling time was 15 minutes. The speed of the containers and main disc was 500 and 1000 rpm respectively. Cylindrical samples, 10 mm in diameter, were elaborated by closed die pressing. A compacting pressure of 600 MPa was employed. The samples were then sintered in vacuum ($10^{-5}$ torr) for 30 minutes when the sintering temperature was 1050 °C.

The samples were then investigated from the tribological and microstructural point of view. The average friction coefficient and the wear rate were determined against a cast iron plate by a pin-on-disc method on a laboratory tribometer. The speed of the plate was 40 rpm while the loading charge was 10 N. Tests were performed at room temperature under dry conditions. SEM, EDX and XRD analyses were also performed. The SEM images were obtained with a JEOL JSM 5600 LV electronic microscope. The EDX spectra were obtained at the X-ray analysis – Oxford Instruments. The XRD analyses were carried out with a Shimadzu XRD-6000 Diffractometer. A Kα-copper X-ray tube with $\lambda = 1.5418$ Å was used. The measuring angle was ranged between 20 and 100 °. The scanning speed was 2 °/min.
Results and discussions

Tribological characterization
Tribological characterization of the elaborated samples implied the determination of friction and wear characteristics. First the average friction coefficient was determined in order to characterize the composites from the friction point of view. The fluctuation of this parameter dependent on the barium carbonate content is graphically given in Fig. 1. The addition of 2 wt% barium carbonate determined the increase of the friction coefficient by almost 45%. Further, increasing the barium carbonate content up to 8 wt% caused the decrease of friction parameter. A possible explication for the increase when adding barium carbonate might be the appearance in the sintered structures of the ceramic component. However 2 wt% barium carbonate seems to represent the saturation for this element that determines the improvement of the friction coefficient.

![Fig. 1. The fluctuation of average friction coefficient dependent on the barium carbonate content](image1)

![Fig. 2. The influence of barium carbonate content on the wear rate of a Fe-Cu-graphite-Ni material](image2)
Concerning the wear characteristics, the wear rate for the four friction composites was determined. Fig. 2 shows the fluctuation of this parameter in terms of barium carbonate content. Adding 2 to 6 wt% barium carbonate determined the decrease of the wear rate with about 20%. Instead, increasing the barium carbonate content to 8 wt% caused the increase of wear parameter. It seems that barium carbonate acts as a lubricant component for the studied sintered iron-based friction composites.

The average friction coefficient/wear rate ratio was determined in order to establish the composite characterized by the best tribological behavior. The variation of the mentioned ratio dependent on the barium carbonate is shown in Fig. 3. A significant increase of the average friction coefficient/wear rate ratio was marked out when 2 wt% barium carbonate were added. It is due to the increase of friction coefficient with reducing the wear rate. Increasing the barium carbonate content from 4 to 8 wt% caused instead a slight decrease of the determined ratio. The Fe-10%Cu-7%graphite-12%Ni-BaCO₃ composite containing 2 wt% barium carbonate is characterized by the best tribological behavior based on the average friction coefficient/wear rate ratio calculated for each material.

![Graph showing the fluctuation of average friction coefficient/wear rate ratio dependent on the barium carbonate content](image)

**Fig. 3.** The fluctuation of average friction coefficient/wear rate ratio dependent on the barium carbonate content

**Structural characterization**

Several EDX analyses carried out with a X-Ray Analysis – Oxford Instruments were performed in different areas of the obtained materials in order to control the presence of the initial elements. These analyses confirmed both the presence of all elements added when elaborating the composites (iron, copper, nickel, barium and oxygen) and the conservation of their proportion (Fig. 4).

Three areas - different in aspect and color - were identified on the SEM images. Fig. 5 shows a SEM image of the FCBC 65 sintered material. The grey areas (1) of irregular shapes represent the matrix formed of iron grains wherein graphite and nickel diffused. A solid solution of Fe (α) alloyed with nickel, (Fe, Ni), pearlite and alloyed ferro-cementites mechanical mixtures constitute the matrix. The black areas (2) were identified to represent the free graphite that remains at the grains boundaries of the sintered structures. The light areas (3) were determined to represent the segregated traces of barium carbonate that were highlighted on the XRD patterns. The increase in quantity of barium carbonate traces was noticed when the barium carbonate added at the elaboration of the researched materials was increased. A decrease of the free graphite content was noticed when increasing the barium carbonate content.
Fig. 4. EDX spectrum of the FCBC 65 sintered composite

Fig. 5. SEM image of the FCBC 65 sintered materials
Note: 1 - matrix; 2 - free graphite; 3 - traces of barium carbonate
The distribution of formed phases in the sintered sample structure has an important influence on the tribological properties of friction composites. The distribution maps of the initial elements (iron, copper, graphite, nickel, barium and oxygen) for the FCBC 65 composite are presented in Fig. 6. A homogenous distribution of copper and oxygen in composite structure was noticed. Some of graphite content was localized at grains boundaries as free graphite. Areas where nickel and barium segregated were marked out. The iron was distributed all over material structure, excepting the barium segregated areas.

![SEM image and the distribution maps of the initial elements in the FCBC 65 sintered composite](image)

Fig. 6. SEM image and the distribution maps of the initial elements in the FCBC 65 sintered composite

The XRD pattern for the FCBC 65 composite both in powder and sintered state is given in Fig. 7. The XRD analyses for the material in powder state highlighted the presence of all peaks
corresponding to the initial materials (iron, copper, graphite, nickel and barium carbonate). The presence of the nickel alloyed ferrite (Fe, Ni) and of copper-based solid solution with nickel (Cu, Ni) as well as the presence of the free graphite was marked out on the XRD pattern of the sintered composite. Some traces of both nickel and barium carbonate were still noticeable in the structure of the sintered iron-based composites.

Fig. 7. XRD pattern of the FCBC 65 material
Note: □ represents the traces of nickel while ♦ represents the traces of barium carbonate

Conclusions
Concerning the influence of barium carbonate content on the tribological characteristics of an iron-based friction composite, we can conclude that the presence of barium carbonate in the Fe-10%Cu-7%graphite-12%Ni material has an important influence on its tribological properties. The addition of 2 wt% barium carbonate led to the increase of the friction coefficient by almost 45 %, this composite being characterized by the highest value of the friction parameter. Adding barium carbonate in the Fe-10%Cu-7%graphite-12%Ni composite determined the decrease of the wear rate and the improvement of the wear resistance respectively, the smallest value of the wear rate being characteristic to the composite containing 6 wt% barium carbonate. Regarding the average friction coefficient/wear rate ratio, the Fe-10%Cu-7%graphite-12%Ni-BaCO₃ composite containing 2 wt% barium carbonate presented the highest value of the mentioned ratio and, implicitly, the best tribological behavior. Nickel alloyed ferrite, pearlite, alloyed ferro-cementites mechanical mixtures, free graphite as well as traces of nickel and barium carbonate were identified in the sintered structure of the researched materials. Future research will attempt to improve the tribological properties of the studied iron-based friction composites by adding different ceramic components such as alumina, titanium dioxide and so on.
References

[1] V.V. Merie, V.C. Cândea, C. Bîrleanu, P. Păşcuţă, C.O. Popa, The influence of titanium dioxide on the tribological characteristics of a Fe-based friction composite material, J. Compos. Mater. 48 (2014) 235-243.

[2] V.V. Merie, V.C. Cândea, C.O. Popa, The influence of nickel content on the properties of Fe-based friction composite materials, Metalurgia International XVI (2011) 93-96.

[3] V. Merie, M. Pustan, C. Bîrleanu, Nanocharacterization of some Fe-based friction composites. ACTA Technica Napocensis. Series: Applied Mathematics and Mechanics 56 (2013) 709-714.

[4] W. Österle, H. Kloß, I. Urban, A.I. Dmitriev, Towards a better understanding of brake friction materials, Wear 263 (2007) 1189–1201.

[5] C. Ferrer, M. Pascual, D. Busquets, E. Rayon, Tribological study of Fe-Cu-Cr-graphite alloy and cast iron railway brake shoes by pin-on-disc technique, Wear 268 (2010) 784–789.

[6] S.N. Namazov, Properties and structural characteristics powder sintered materials on the basis of iron, Savez Inzenjera Metalurgije Jugoslavije (2002) 111-119.

[7] M. Asif, K. Chandra, P.S. Misra, Development of iron based brake friction MMC used for military aircraft application by a new P/M route, Journal of Minerals & Materials Characterization & Engineering 10 (2011) 693-705.

[8] C.S. Ramesh, C.K. Srinivas, Friction and wear behavior of laser-sintered iron-silicon carbide composites, Journal of Materials Processing Technology 209 (2009) 5429-5436.

[9] N.M. Talijan, D.S. Trifunovic, D.D. Trifunovic, The influence of different iron powders on the friction properties of sintered friction materials based on iron, Materials Letters 46 (2000) 255-260.

[10] T.K. Bandyopadhyay, S. Chatterjee, K. Das, Synthesis and characterization of TiC-reinforced iron-based composites, Journal of Materials Science 39 (2004) 5735-5742.

[11] S. Shamsuddin, S.B. Jamaludin, Z. Hussain, Z.A. Ahmad, Characterization of Fe-Cr-Al$_2$O$_3$ composites fabricated by powder metallurgy method with varying weight percentage of alumina, Journal of Physical Science 29 (2008) 89-95.

[12] A.I. Dmitriev, W. Österle, H. Kloß, About the influence of automotive brake pad composition on frictional performance. Results of nano-scale modeling, Nanosystems: Physics, Chemistry, Mathematics 2 (2011) 58-64.

[13] M. Asif, K. Chandra, P.S. Misra, Development of iron based brake friction material by hot powder preform forging technique used for medium to heavy duty applications, Journal of Minerals & Materials Characterization & Engineering 10 (2011) 231-244.