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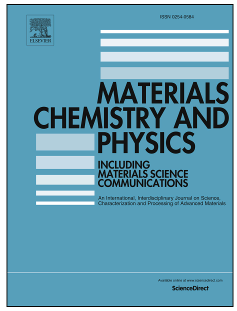

Enhancing composite materials through fly ash reinforcement in powder metallurgy

Clayton André Oliveira da Motta, José de Souza, Vinícius Martins, Lirio Shaeffer, Elton Gimenez Rossini, Luciano Volcanoglo Biehl, Diego Augusto de Jesus Pacheco, Carlos Otávio Damas Martins, Jorge Luis Braz Medeiros

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CRediT authorship contribution statement

Clayton André Oliveira da Motta: Research idea, Conceptualization, Methodology, Investigation, Writing – original draft, Writing - review & editing, Project administration. **José de Souza:** Research idea, Conceptualization, Methodology, Investigation, Writing – original draft, Writing - review & editing. **Vinícius Martins:** Research idea, Conceptualization, Methodology, Investigation, Writing – original draft, Writing - review & editing. **Lirio Shaeffer:** Methodology, Investigation, Writing – original draft, Writing - review & editing. **Elton Gimenez Rossini:** Methodology, Investigation, Writing – original draft, Writing - review & editing. **Jorge Luis Braz Medeiros:** Methodology, Investigation, Writing – original draft, Writing - review & editing. **Luciano Volcanoglo Biehl:** Methodology, Investigation, Writing – original draft, Writing - review & editing. **Diego A. de J. Pacheco:** Investigation, Writing – original draft, Writing - review & editing. Supervision. **Carlos Otávio Damas Martins:** Investigation, Writing – original draft, Writing - review & editing.

TITLE PAGE

Enhancing composite materials through fly ash reinforcement in powder metallurgy

Prof. Clayton André Oliveira da Motta
Postgraduation Program in Materials Engineering
Engineering School, Federal University of Rio Grande do Sul
Av. Bento Gonçalves, 9500, Porto Alegre, RS, Brazil
Phone: +55 51 33089428
https://orcid.org/0000-0002-2391-816X
E-mail: clayton.motta@yahoo.com.br

Prof. Dr. José de Souza
Department of Research and Industrial Production
Liberato Salzano Vieira da Cunha Technical School Foundation
Inconfidente, 395, Novo Hamburgo, RS, Brasil
https://orcid.org/0000-0001-5888-840X
E-mail: josesouza@liberato.com.br

Prof. Dr. Vinícius Martins
Postgraduation Research and Innovation
Federal Institute of Rio Grande do Sul
Av. Copacabana - Piratini, Sapucaia do Sul - RS, Brazil
Phone: +55 51 34529240
https://orcid.org/0000-0002-0301-2723
E-mail: vmartins@sapucaia.ifsul.edu.br

Prof. Dr. Lirio Shaeffer
Postgraduation Program in Materials Engineering
Engineering School, Federal University of Rio Grande do Sul
Av. Bento Gonçalves, 9500, Porto Alegre, RS, Brazil
Phone: +55 51 33089428
https://orcid.org/0000-0002-3427-2405
E-mail: schaefer@ufrgs.br

Prof. Dr. Elton Gimenez Rossini
Energy Engineering Department
State University of Rio Grande do Sul
Rua Washington Luiz, 675, Porto Alegre, RS, Brazil
Phone: +55 51 32281731
https://orcid.org/0000-0002-4195-2003
E-mail: elton-rossini@uergs.edu.br

Prof. Dr. Luciano Volcanoglo Biehl
Postgraduation Program in Mechanical Engineering
Engineering School, Federal University of Rio Grande
Av. Itália, s/n - km 8 - Carreiros, Rio Grande, RS, Brazil
Phone: +55 53 32336500
https://orcid.org/0000-0002-5415-5863
E-mail: lucianobiehl@furg.br

Prof. Dr. Diego Augusto de Jesus Pacheco (corresponding author)
Aarhus University, BTECH, Engineering and Technology Division
Birk Centerpark 15, 7400, Herning, Denmark

Phone: +45 87164700
https://orcid.org/0000-0003-4453-7216
E-mail: diego@btech.au.dk

Prof. Dr. Carlos Otávio Damas Martins
Universidade Federal do Sergipe, Department of Science and Materials Engineering
https://orcid.org/0000-0002-2964-362X
E-mail: carlosmartinsufs@gmail.com

Prof. Dr. Jorge Luis Braz Medeiros
Postgraduation Program in Mechanical Engineering
Engineering School, Federal University of Rio Grande
Av. Itália, s/n - km 8 - Carreiros, Rio Grande, RS, Brazil
Phone: +55 53 32336500
https://orcid.org/0000-0003-3468-7632
E-mail: jorge.braz@furg.br

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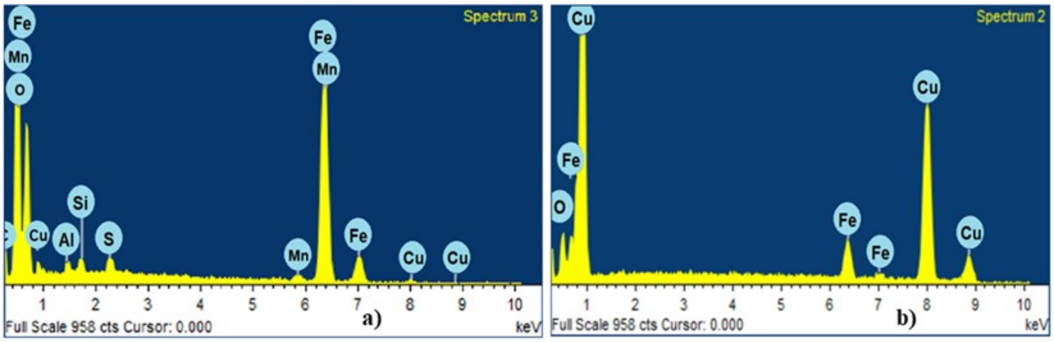
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Enhancing composite materials through fly ash reinforcement in powder metallurgy

Abstract

The utilization of fly ash as reinforcement in composites offers a cost-effective alternative for applications involving ductile metallic matrices. Ash is a by-product of coal combustion in thermoelectric plants, and its use can serve as an environmentally friendly option. Fly ash exhibits characteristics of a ceramic material. However, there are gaps in understanding the mechanical and metallurgical behavior resulting from the interaction between the matrix and the fly ash-based reinforcement. This study aims to develop and evaluate the application of various amounts of fly ash as reinforcement in metal matrix composites through the powder metallurgy process. The process adheres to internationally recognized standards for incorporating ashes into metallic composites. The method involved producing specimens with no fly ash (0FA) and using 5%, 10%, and 15% fly ash (5FA, 10FA, 15FA). The samples were assessed for density, microstructure, microhardness, and wear coefficient using established techniques. Through graphical and statistical analyses, the research examined the impact of reinforcements on the metallic matrix. The results indicate that including fly ash as a composite reinforcement led to a reduction in density with a variance level of F' 17.7. The microhardness values were not significantly affected, with F' 0.46. There was a noticeable improvement in wear resistance for 200 cycles (F' 22.7), 400 cycles (F' 4.7), and 600 cycles (F' 0.58). In conclusion, employing composites reinforced with fly ash of varying compositions is feasible, using highly applied compressive pressures and the powder metallurgy process.

Keywords: powder metallurgy; fly ash; mechanical properties; mechanical behavior; metallic composites.

1. Introduction

To enhance economic and environmental efficiency while promoting sustainability in manufacturing processes, there is a growing interest in utilizing residues as raw materials in powder metallurgy. For instance, studies have explored the use of AISI D2 steel grinding mud, which contains silicon carbide. This mud can serve as a reinforcement in composites or be integrated with steel by-products, enabling recycling possibilities [1, 2]. Such approaches offer the potential for cost-effective and environmentally friendly manufacturing practices.

Understanding the mineralogical, micromeritic, and rheological properties of various materials, such as powdered Fe, powdered SiC, powdered Al, powdered WC, coal fly ash, steel turning chips, steel grinding chips, and aluminum saw chips, is crucial. Research has shown that residual metallic chips can be directly recycled using powder metallurgy, eliminating the need for time-consuming ball milling operations. By quantifying rheological attributes such as the Hausner ratio, Carr index, and angle of repose, it is possible to assess the feasibility of using these manufacturing products as reinforcements in metal matrix composites (MMC) [3, 4].

However, the relationship between the utilization of fly ash as reinforcement in metal matrix composites (MMC) and its impact on mechanical and metallurgical behavior has not been sufficiently elucidated. This research addressed this gap by developing and testing a method to analyze the influence of Fly Ash (FA) insertion in Fe-Cu-C alloys MMC manufactured through Powder Metallurgy (PM). The addition of copper in the Fe-Cu-C composites aimed to enhance material densification during liquid-phase sintering and reduce voids by minimizing dendrite formation. The mechanical properties were improved by introducing fly ash as reinforcement [5]. Fly ash is a solid by-product derived from coal combustion plants and consists of spherical particles containing silica, aluminum, iron, calcium, and titanium oxides ranging from 1 to 100 µm in size [2]. Utilizing fly ash residues to develop novel and valuable materials through the PM method is highly desirable [6].

PM minimizes material losses and production costs by reducing the need for subtractive manufacturing processes [5,6]. PM offers advantages such as fabricating complex geometries and sizes at lower temperatures and using cost-effective machinery with lower energy consumption. Compared to conventional methods like casting and machining, PM requires minimal post-processing. The process involves powder mixing, compaction, and sintering [5,6]. Powders can be pure or pre-alloyed elements which, together with additives, are mixed using mechanical mixers. Compaction is carried out using machined dies with high tolerances and applying uniaxial loads using hydraulic or mechanical presses. The fundamental objective of compaction is to obtain a green compact with sufficient mechanical strength to withstand other handling operations before sintering. Sintering is carried out in a controlled atmosphere for atomic bonding with volumetric shrinkage. Densification occurs by the diffusion of atoms at temperatures lower than the melting temperature of the materials, improving the metallurgical and mechanical properties of the materials [7]. In addition, working with powdered materials allows for obtaining different chemical compositions [5]. In this sense, specifically, our research is focused on applying Fe – Cu – C – FA composites to manufacture mechanical seals in Figure 1.

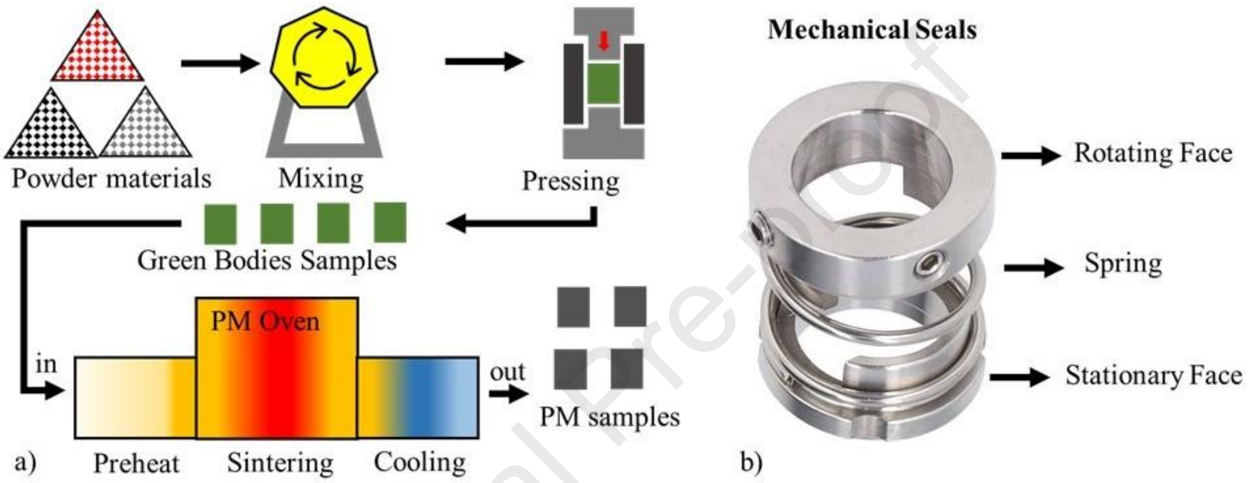

Figure 1. PM process in a) and the manufacture of mechanical seals in b).

The mechanical seals are cylindrical parts that aim to eliminate and/or prevent fluid leaks, preventing their passage along the interface of two radial annular surfaces. They are used in rotating equipment such as centrifugal pumps and other equipment in the most different industrial sectors such as automotive, textiles, food, oil & gas, pulp and paper. These components are generally manufactured through casting and/or forging, followed by machining for final finishing.

Previous literature has shown that an increase in the concentration of FA particles can lead to a shift in fracture mode from a combined ductile-brittle mechanism to a predominantly brittle mechanism, which can compromise the performance of structural parts [8]. Early studies focused on evaluating the impact of FA additions on the properties of iron powder (99.5%) [2,3,4]. Results indicated that FA, with its spherical geometry and small particle size, effectively filled voids, resulting in improved densification of both green bodies and sintered alloys. The highest densification values were achieved with an alloy containing 10% FA sintered at a maximum temperature of 1150 °C.

Another study evaluated the influence of sintering temperature (900, 950 and 1000 °C) on the properties of a copper alloy with 30%wt addition of FA. The results indicated the possibility of including FA as reinforcement in copper alloys. Additionally, the porosity values found showed that melting is predominant over sintering for temperatures above 950 °C alloy [5]. Previous works also evaluated the effect of adding FA on the metallurgical properties of cast alloys of the type A354 (Al – Si – Mg – Cu) used to manufacture cylinder heads. Working with additions of 5, 10 and 15% of FA, it was demonstrated that the small additions of FA (5 and 10%) did not compromise the mechanical properties concerning the base material [8].

An important distinction between the present study and much of the extant literature is that, specifically, we sought to evaluate the influence of adding FA on the tribological properties of the sintered Fe-Cu-C alloy. Therefore, compositions containing 5, 10 and 15% of FA mass additions were tested in our study. The materials were evaluated through microstructural analysis, density, hardness and wear tests, where the properties were compared with the alloy without FA.

Our findings demonstrate that including up to 10% of FA does not compromise the performance of the materials, as measured by density and coefficient of friction, particularly in analyses with a few cycles. This contradicts previous literature and highlights the effectiveness of our evaluated efforts. For the 15% FA composition, the increase in porosity and wear rate weakened the material. This article contributes to developing new technologies that optimize the utilization of inputs and waste while maintaining the processability and mechanical performance of sintered materials. The study enhances our understanding of the effect of using low-cost carbon-based ash as reinforcement in metal matrix composites and the diffusion conditions in the substrate.

2. Methodology

The selection of standard materials for the study was based on their physical, chemical, and mechanical properties, as well as their potential for environmental recycling. Atomized iron powder with a purity of 99.5% and particle size ranging from 45 to 212 μm was obtained from an industrial company. Coal fly ash used in the research was sourced from the carboniferous mining regions in South America, with a granulometry of up to 610 μm. The chemical composition of the materials is presented in Table 1, indicating the input parameters for the analysis.

For the evaluation of the influence of the addition of fly ash on the physical and mechanical properties of a Fe – Cu – C alloy, sintered by powder metallurgy, four groups of samples were prepared to contain different insertions of 0 (0F - Base), 5 (5FA), 10 (10FA) and 15% (15 FA) in the mass of fly ash (composites) according to Table 1.

Table 1. Chemical compositions of the matrix and reinforcement based on fly ash.

Materials	Composition [% mass]			
Composites	*% Fe*	*% Cu*	*%C*	*% Fly Ash*
0FA - Base	65	34,8	0,2	0
5 FA	55	34,8	0,2	5
10FA	55	34,8	0,2	10
15 FA	50	34,8	0,2	15

The experimental process involved the preparation of eight cylindrical specimens measuring 6 mm in height and 13 mm in diameter using a specific chemical composition. The preparation of the samples was carried out in three stages. The first stage involved the characterization and preparation of the powders, including electrolytic copper powder, atomized iron powder, graphite powder, and zinc stearate as a lubricant. To incorporate coal fly ash, small amounts were added as a replacement for the iron powder in the composition. Finally, the mixtures were thoroughly blended using a double-cone mixer.

In the subsequent stage of compression of the green bodies, the specimens were compacted with a manual hydraulic press model P30ST. Eight uniaxial load cycles were applied during the compaction process. The samples were sintered in an oven Sanchis model S1, with an inert atmosphere based on argon. A sintering temperature of 1150 ℃ and a cooling rate of 10 ℃/min were used.

Next, the samples were submitted to tests to determine their properties, such as apparent density and sintered density, microhardness, microstructure, dimensional control, and abrasion resistance with the tribological test. The density was measured using the Archimedes method, with the aid of an analytical scale from Urano, model 1000/0.1l.

The metallographic preparation followed the ABNT NBR 15454 (2007) standard’s provisions, involving sanding, polishing with diamond paste and chemical etching. For visualization, a Zeiss AG – EVO 40 SEM model was used. Hardness tests were performed on the Vickers scale according to the ISO 6507:2018 using a Shimadzu micro-durometer (HMV 2 T) equipment. The specimens compacted at 800 MPa were tested, using a load of 50 g, for 10 seconds at 10 points per sample.

Wear tests were carried out with the aid of a CETR tribometer using the ball-on-plate method. First, grinding was performed with reciprocal linear movement by an alumina sphere with a diameter of 7.75 mm, load of 5N, and frequency of 1 Hz for 15 minutes, according to the procedure outlined in ASTM G99-2017. Next, the samples were analyzed in the CETR Pro500 3D profilometer to verify the dimensions of the tracks.

The green density and density after sintering are critical phases, mainly in generating mixtures using ceramics (FA) in PM. Fly ash composites tend to generate low-density products [3, 4]. Using cylindrical samples of low volume and area allows a quick analysis of their density using the Archimedes method. Optical Microscopy (MO) and Scanning Electron Microscopy (SEM) are necessary for the analysis techniques after the sintering step. SEM allows the high-quality analysis of organic and inorganic microstructures without the need for fragmentation of the specimens to be analyzed [9, 10].

Quantitative analysis of the composite can be conducted using SEM. Electron Dispersion Spectroscopy (EDS) is utilized to determine the distribution of each element within the compound's mixture, validating the product and certifying the percentages of each element within the matrix. Since these composites contain a ceramic element in their matrix, including fly ash allows for assessing its potential for wear, both as a cause and a consequence [11, 12]. Wear tests typically require fixation devices for the samples being tested. However, for this research, the generated samples' format and dimensions eliminated the need for such devices. The analysis of the worn regions is best performed in conjunction with the numerical results. Wear profiles are presented through 2D and 3D graph plotting, and the variation in color in the 3D graphic allows for a visual assessment of the prompt wear action on the samples.

Finally, the method includes the Vickers microhardness test to evaluate the performance. This test allows for a more accurate assessment of regions with high concentrations of each element or group of components formed during sintering, reducing common porosity in PM-manufactured products [13]. The study contributes to understanding the impact of using low-cost carbon-based ash as reinforcement in metal matrix composites and the diffusibility condition in the substrate. The collected data were analyzed using a significance level of 5% through ANOVA. The tests were designed as factorial experiments. ANOVA is a parametric test that assumes a normal distribution of values (null hypothesis).

3. Results and Discussion

In the first stage, the preparation and mixing of a ternary composite showed homogeneity regarding the chemical compositions 0FA, 5FA, 10FA, 15FA. The sintered samples were visually evaluated as free of cracks or surface erosion [14]. Proper data collection and analysis must select a sufficient amount of sintered parts [15]. For this research, 8 (eight) samples of each compound generated were used for the tests and subsequent analysis [10,11,12,13].

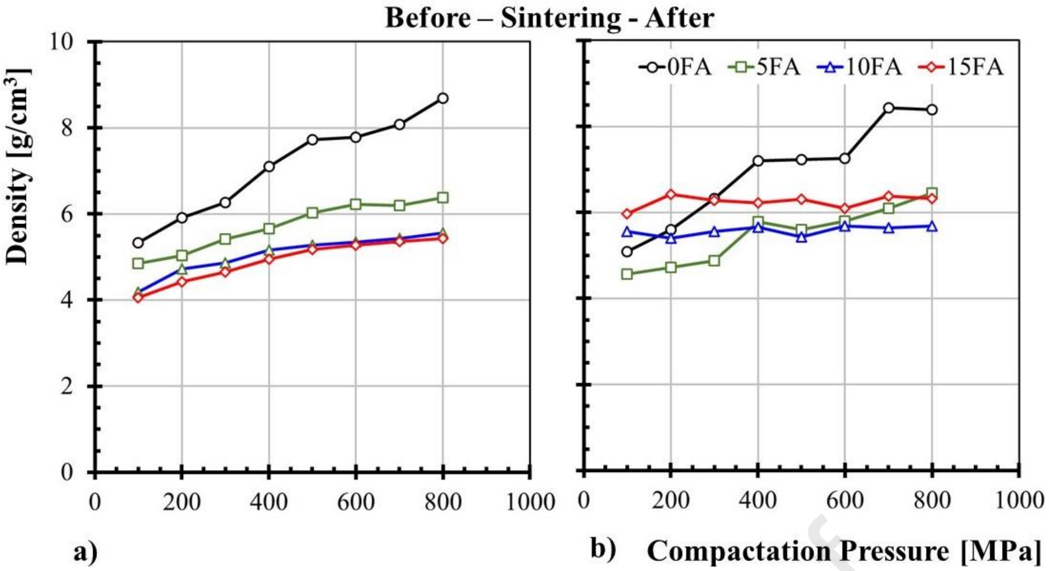

Figure 2. Density before a) and after sintering b).

In the second stage, the density of the developed composites is verified. The values found by the Archimedes test before and after sintering can be seen in Figure 2. After sintering, the FA sample had a lower density, demonstrating a better apparent improved matrix-reinforcement interaction. According to standard test methods for the density of sintered PM products using the Archimedes principle, composites with ash should have average densities close to the density of the composite without ash [14, 15].

SEM and EDS analysis of Fe-Cu-C and Fe-Cu-C-Fly ash composites were performed according to NBR 13284: 1995 [17], NBR 15454: 2007 [18] and ASTM E1382: 97 [19]. The results are shown in Figure 3. Sample preparation in the application of the method involved careful metallographic preparation [20, 21]. The size of the analyzed region and the depth of the electron beam occurs preliminary to the application of the test. SEM enabled the analysis of the particle size of the constituent elements. Using the scanning electron microscope (SEM) and the EDS microprobe, it was possible to qualitatively verify the elements present in the matrix and reinforcement with fly ash, as shown in Figures 3 (SEM) and 4 (EDS).

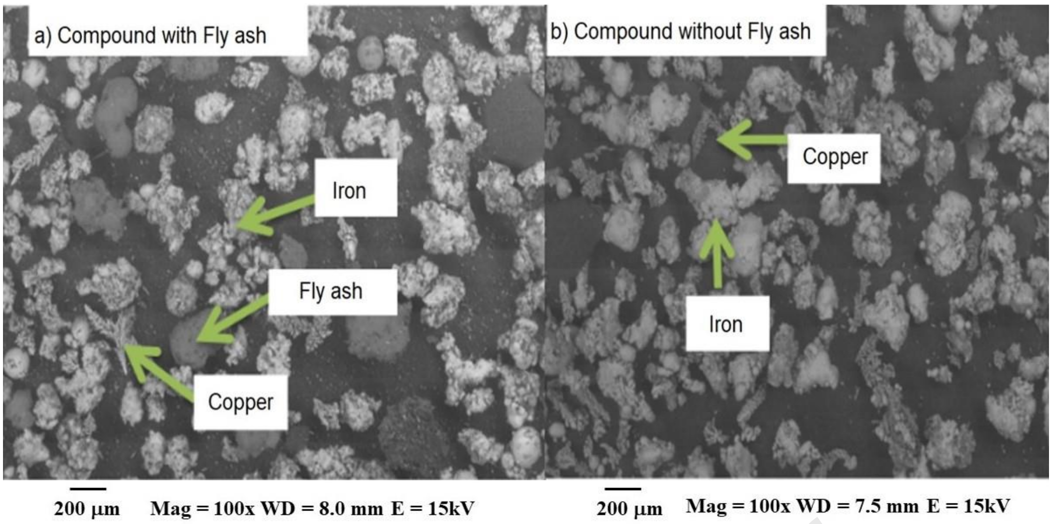

Figure 3. Matrix and chemical distribution of composites - sintered sample: a) Compound A with ash. b) Compound without ash.

The analysis identified the particle morphology of copper, iron, and fly ash in the formulation. Iron particles appeared as irregularly shaped agglomerates with a white color, while copper powders exhibited a feather-like form. FA, on the other hand, displayed a predominantly spherical shape, distinguishing it from the other elements.

Each chemical element belonging to the sintered matrix behaves differently, and it is also necessary to analyze its shape and organization in the matrix. This behavior is associated with its condition of interstitial diffusion, element substitutions and the associated kinetics. The sintering atmosphere is also highly relevant in the sintering process. The imbalance of these conditions can be monitored to avoid deleterious effects on the substrate and surface [5,6,8]. Both analyses must take place on the same image collected by the test instrument, with the analyzed region's magnification and the scale for analyzing the adjusted particle size. At this stage occurs an analysis of the neck formation process and the iteration between the particles in the sintering process by observing the collected image.

The qualitative and quantitative analyses were performed using the EDS microprobe coupled to the SEM. The EDS graph depicted the distribution of fly ash within the ternary composites, as illustrated in Figure 4. These analyses aimed to examine if there was ash diffusion during the sintering process and to identify the residual ceramic elements present in the resulting matrix. Additionally, the morphology and interactions of the formed composites were evaluated. The equipment that once provided micrographic data must now be adjusted to provide a qualitative assessment of the analysis region. The tested region may be the same as the SEM region. However, removing the samples from the equipment for surface cleaning occurs, thus allowing a test without contaminants.

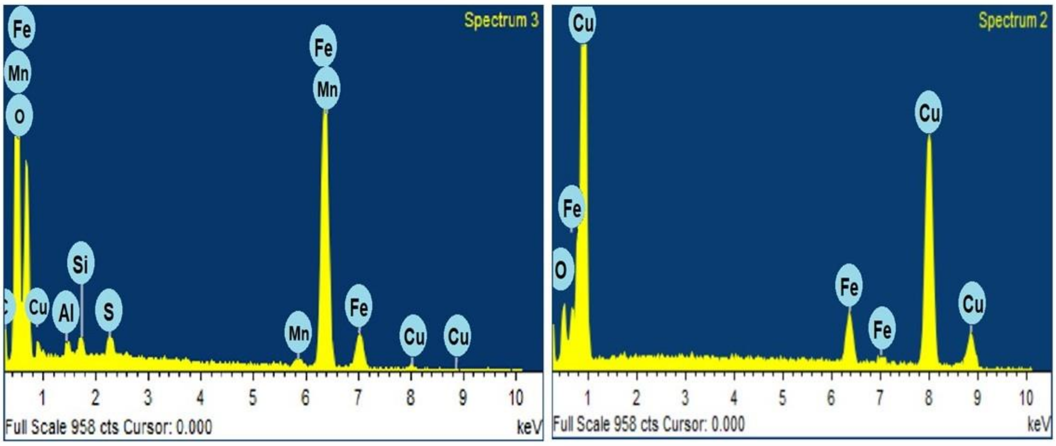

Figure 4. EDS of the composites of sintered samples. a) Composite A with fly ash. b) Composites without fly ash.

These results in Figure 4 indicated the presence of ceramic elements in the matrix of ternary composites in the EDS of fly ash composites, justifying the correct sintering of the tested samples.

In the fourth stage of the method, the samples were submitted to a tribological wear test using a CETR tribometer. This test produces wear due to the reciprocal linear movement promoted by an alumina sphere with a diameter of 7.75 mm. A parameter in applying this test occurs a load of 5N for 15 minutes with a speed of 1.0 Hz (Figure 5). In addition, a profilometry analysis of the worn region happens as part of the wear test analysis. Therefore, Profilometry was utilized as a complementary analysis to wear so that a profile of the exposed region happens. This analysis enabled studying the influence of ceramics on the wear resistance of bodies sintered with ash and comparing them to the compound without ash.

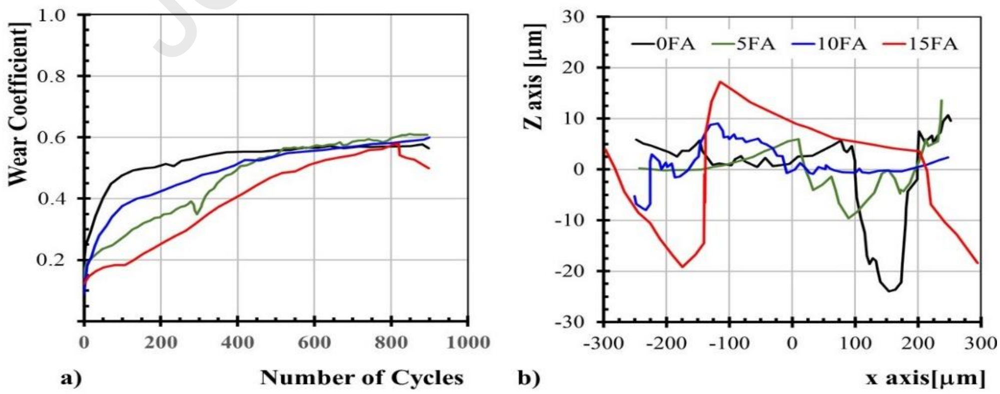

Figure 5. a) Composite wear test with different cycles. b) In effect on the X and Z axes.

The tribological wear test should evaluate the impact of the ceramic elements present in FA on sintered composites. Figure 6 shows the profile of the worn region in 2D format. In the horizontal axe (x), the length of the cap radius appears in (µm) and in (Z), the depth (Z) of the cap radius in (µm), generated in the region affected by the wear test of the compost with fly ash.

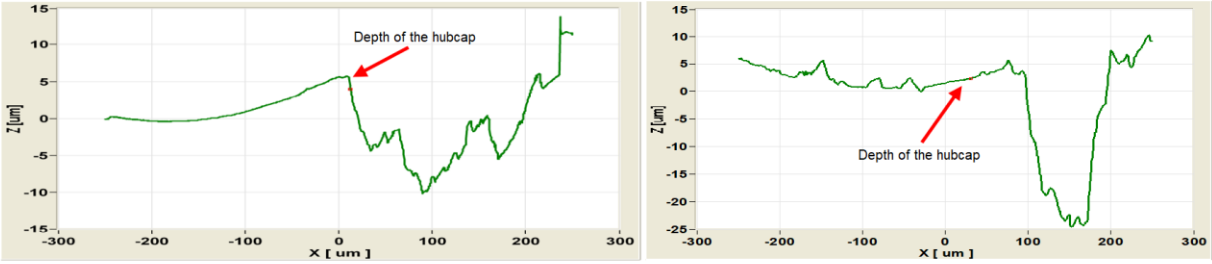

Figure 6. 2D profile of the worn region of the fly ash samples region with more accentuated wear and formation of valleys.

Another important outcome observed refers to the profilometric test. Figure 7 shows the 3D profilometric test results observed in the red region with homogeneous wear and in the blue regions, where valleys formed by the ball on the plate can be observed.

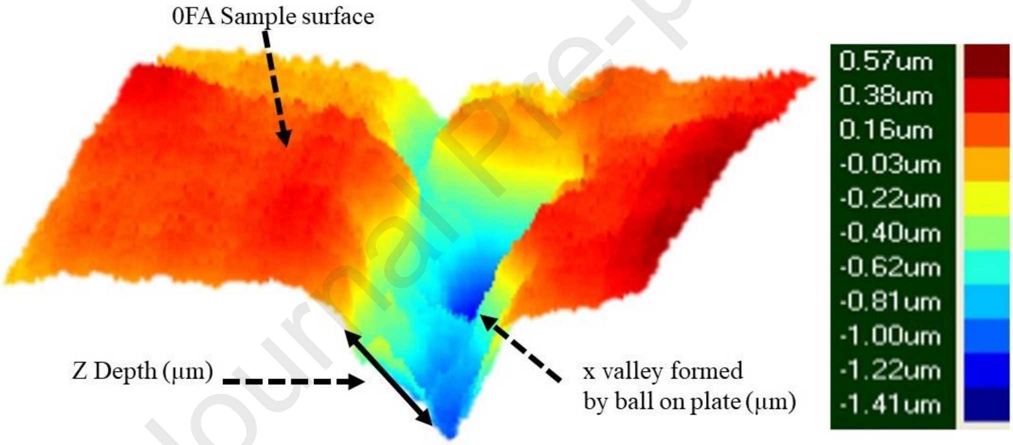

Figure 7. Profilometric test results in 3D. In the red region with homogeneous wear. Blue region with more accentuated wear and valley formation.

In the fifth and last stage of the applied method, we analyzed the behavior of the mechanical properties of hardness between the composites of different percentages of ash and the compound without ash. The test to verify the hardness of sintered composites that can offer granular hardness behavior with greater assertiveness is the Vickers microhardness test [22].

In Vickers microhardness, the region of analysis must be the grain’s face hardened in sintering and not the natural voids (pores) of the process. The samples must be cut by tools that do not provide heat to the analysis face. The samples’ section must occur by manual tools such as band saws or others without local heat addition. It is unnecessary to perform cold or hot inlay of the tested section, and it can be positioned directly on the load application table. Table 2 shows the average results of 10 measurements observed by Vickers microhardness.

Table 2. Vickers microhardness in samples of composites 0FA (metal), 5 FA, 10 FA, and 15 FA (without fly ash).

Composites Samples	**Average results (HV1)**
0FA - Base	105
5FA	129
10FA	130
15 FA	102

Materials with low, medium or high porosity levels must evaluate the mechanical properties of hardness and the Vickers microhardness test [19, 20]. As these are sintered composites with porosity, more reliable hardness verification was necessary to evaluate a micro-region. The micro-region chosen to analyze the Vickers microhardness in the samples was where there is a predominance of iron, iron-copper, and dissolved fly ash. Copper undergoes sintering in the liquid phase acting as a capillary element, closing pores, and promoting more excellent intermolecular bonding [25,26]. This phenomenon will attenuate the ash's low intermolecular bonding, which is a ceramic element and will promote better mechanical properties results than sintered composites without their addition.

We observed that the microhardness value changes because of the presence of FA in the composites. The experimental results show that FA increases the microhardness, as observed during the fifth stage. Another aspect related to the analysis of the third and fifth stages is that samples with fly ash have more resistance to friction than samples without ash. As a result, the ash compound's coefficient and friction observed were higher, which can be crucial in specific industrial applications.

Finally, Figure 8 presents the results of the analysis of the variance values of the samples regarding density, microhardness and wear resistance. The checks regarding the wear coefficient were carried out considering wear cycles applied at 200, 400 and 660.

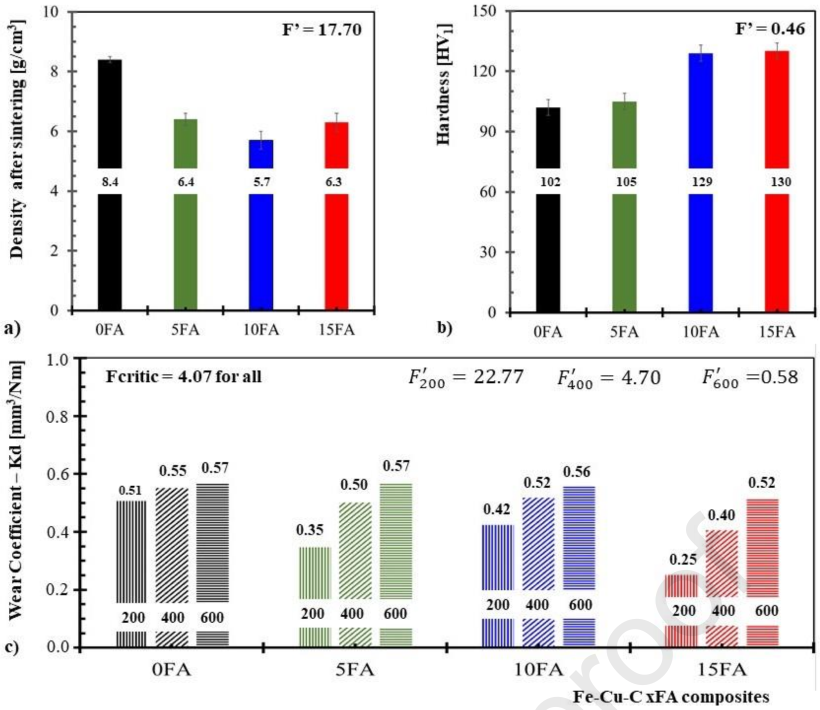

Figure 8. Analysis of variance of the results obtained, density a), microhardness b), and wear coefficient in c).

Figure 8 illustrates the impact of incorporating FA on the physical and tribological properties of the Fe-Cu-C composite. The analysis in c) was conducted for 200, 400, and 600 cycles, as indicated in the columns. The results indicate that FA significantly influences the density after sintering (Figure 7a). This can be attributed to the lower density of FA and the increased volumetric content of FA within the composite.

Along with the density variation, the inclusion of FA also enhances the microhardness of the materials, although no statistically significant difference is observed (Figure 8b). Regarding the wear studies with different cycle numbers (200, 400, and 600 cycles), Figure 8c demonstrates that the increase in FA content had a more pronounced effect in the 200-cycle condition (F' = 22.77). The significance remains, however, with lesser impact for 400 cycles (F'= 4.70) and has no impact for cycles with high wear load (600) (F'= 0.58). These results indicate the possibility of applying composites with the addition of 10% futile ash in the composition. Among the different composite samples, the one with 10% fly ash (10FA) exhibited superior performance in terms of density, microhardness, and tribological applications compared to the 0FA sample. Additionally, in the 400-cycle condition, the 15FA samples demonstrated lower wear coefficients compared to the 0FA, 5FA, 10FA, and 15FA cycles. However, at higher cycles (600), the wear resistance values were diminished due to erosion and surface degradation effects on the samples [8,11,27,28].

4. Conclusions

From the results and discussions, it was concluded that:

- Composites with fly ash exhibit similar properties to ashless composites.
- Micrographic results demonstrate adequate sintering of copper with iron, with diffusely present iron and minimal copper in the investigated ash. The ash investigated in the EDS showed an excellent amount of diffusely present iron, but with very little copper in this situation.
- The developed method shows that the mechanical resistance and dimensional shrinkage properties of composites are similar to the unreinforced matrix.
- The results demonstrated significant changes in the density of composite samples with FA reinforcement.
- After sintering, the FA sample has lower density, indicating improved matrix-reinforcement interaction.
- The decrease in the coefficient of friction contributed to the improvement of the tribological properties of samples with fly ash.
- The addition of fly ash increases microhardness without statistically significant variation.
- Using fly ash percentages above 5% as reinforcement is a viable alternative for metallic matrix composites.
- The 5FA, 10FA, and 15FA composites perform best in tribological applications compared to the 0FA base material. The 10 FA sample had the best result in relation to density, microhardness and in tribological applications, when compared to the 0FA sample.
- Composites with higher ash percentages exhibit regions with more inclusions.
- Due to the presence of impurities like SiO2 and Al2O3, higher amounts of FA can lead to the formation of porous regions. As a result, the 10FA composite exhibited better performance.

Declaration of interests:

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Highlights

Fly ash composites show comparable properties to ash-free composites.

Mechanical strength and dimensional stability of composites resemble the unreinforced matrix.

Fly ash exhibits lower post-sintering density, indicating enhanced matrix-reinforcement interaction.

Fly ash content above 5% as reinforcement is a feasible option for metallic matrix composites.

The research contributes novel insights to the field of powder metallurgy technology.

Declaration of interests

☒ The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

☐The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: