Aspects regarding the correlation between the physical-mechanical and tribological characteristics of composites materials reinforced with carbon fibers

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Abstract. The purpose of this paper is to highlight a number of factors that influence the physical-mechanical and tribological characteristics of sintered composite materials. Such factors are grouped generally in two categories: technological parameters (pressure compacting, sintering temperature, sintering duration, heat treatment) and the receipt of sintered composite materials. In this paper is presented a program of experiments developed both in composite materials sintered polymer matrix (non-metallic) and in the metal matrix (e.g., Al) which was prepared in advance a methodology original production and research for this particular type of materials. The experiments have focused development and testing of a number of 14 polymer composite and 5 composite sintered Al base, in both situations armed with carbon fiber in various forms. Tribological tests followed the establishment of the coefficient of friction and wear rate of the sliding speed at the constant values \( v = 7.2 \, \text{mm/s} \) and the normal load \( N = 8 \, \text{daN} \) and for different orientations of the fibers to the direction of sliding: normal (N type), parallel (P) and antiparallel-perpendicular (AP type).

1. Introduction

Materials for engineering applications can be divided in the following main categories: metals and alloys, ceramics, glass, polymers, elastomers and textile composites [1]. Composite materials have appeared from the need to optimize the properties depending on the followed purpose. Starting from the “cooperation” of component form the composite material so that it can be found only qualities of the components and their deficiencies to be compensate, it can be considered the synergistic properties of composite materials [2,3].

Composite materials represent the latest class of materials. Are modern materials, advanced, that performance exceed in many ways the traditional materials. Production of composite caused the developing of many specific technologies supporting the development and modernization of the modern industrial sectors: the automobile, aeronautics, divers electronic equipment biomaterials, naval, civil engineering and many others.

Matrix composites are very encountered with matrix from alkyd polyester, epoxy resin and vinyl ester resins, reinforced with fiberglass, carbon, boron, silicon carbide and polyethylene [3-5]. Metal matrix composites are very different: with aluminum matrix, magnesium, copper, titanium, alloys and, more recently the super-alloys. These matrices are reinforced with different types of fiber from boron, tungsten oxides, carbides and graphite [6].

Complementary materials fibers type is used as reinforcing elements, serving to take over much of the loads on which is solicited the matrix material. By structure, the fibers may be polycrystalline, monocristalline or amorphous [7].

Carbon fibers have been imposed recently because of the numerous advantages: remarkable mechanical properties (carbon fiber has the highest modulus of elasticity and the highest specific strength of all materials used to produce fiber), low cost, stability at high temperatures, good chemical compatibility reported with the organic matrix and the possibility of using a variety of raw materials [8-10].
2. Analysis of the composite materials sintered with the polymeric matrix
The experiments program afferent to obtain and analysis of the polymeric composite sintered was focused on four clear areas, namely [11]:
- Phenol formaldehyde resins reinforced with short carbon fiber;
- Epoxy bicomponent resins reinforced with carbon short fiber;
- Single-component epoxy resin with modified mono-component reinforced with tape of unidirectional carbon fiber;
- Epoxy resin with modified resin reinforced with carbon fiber texture.

| Phenol-formaldehyde polymer formulations sintered | I          | II          |
|--------------------------------------------------|------------|------------|
| Fiber volume fraction                            | 2.67       | 22.54      |
| (manual shredded)                                |            |            |
| Graphite                                         | 83.84      | 33.43      |
| Resin                                            | 15.79      | 15.82      |
| Graphite big grain (0.08 mm)                      | –          | 30.62      |

Required characteristics
- Density, g/cm³
  - I: 1.712
  - II: 1.816
- Porosity, %
  - I: 4.2±8.25
  - II: 10.10±15.11
- Mechanical strength, daN/mm²
  - I: 0.5±1.8
  - II: 0.7±1.1

Note: The use of the wet treatment (40% nitric acid and a treatment time of 30 min) carbon fibers.
One-component composite resin formulations

| Required characteristics | Alepo 01 | Alepo 02 |
|--------------------------|---------|---------|
| Fiber volume fraction, % | 17      | 17      |
| Hot pressing and curing (180°C, 60 min) |          |         |
| Mechanical resistance: 0.48±2.31 daN/mm² |          |         |
| Coefficient of thermal expansion: 4.01±6.5x10⁻⁶ °C⁻¹ |          |         |
| Hardness: 1.019±2.01 g/cm³ |          |         |
| Porosity: 4.02±21.43 % |          |         |

Note: 1. It is recommended to use the carbon fiber, wet treated 40 ÷ 50% nitric acid with a treatment time of 30 ÷ 50 min
2. The dimensions of the fiber chopping: 2÷4 mm

Two component resin composite formulations

| Required characteristics | Alepo IV | Alepo V | Alepo VI | Alepo 0 | Alepo 5 |
|--------------------------|---------|---------|---------|---------|---------|
| Fiber volume fraction    | 25.5±27.9 | 20.8±22.3 | 24.4±28.4 | 27.5±28.1 | 23.5±24.6 |
| Hardener DEDA            | 12%     | 12%     | 12%     | 12%     | 12%     |
| Cold hardening           | 48.2±49.3 | 34.56±44.9 | 47.28±48.88 | 41.3±43.13 | 32.4±34.14 |
| Hardener DYCY            | 50%     | 50%     | 50%     | 50%     | 50%     |
| Hot pressing and hardening (170 °C) |          |         |         |         |         |

Note: It recommended working with carbon fibers electrochemically treated in electrode potential between 2.1 ÷ 2.7 Ve.s.c. and the load current of 3500 ÷ 4000 C/g or carbon fibers treated wet with nitric acid 30 to 80% and a treatment time of 30 ÷ 60 minutes.

Sintered polymer material formulations

| Percentage fiber | 38.75 | 50.73 | 45.15 | 46.77 | 40.70 | 48.71 | 47.71 | 42.61 | 46.44 | 47.88 |
| Resin           | 40.86 | 25.35 | 27.32 | 30.89 | 31.26 | 42.68 | 29.41 | 23.45 | 36.41 | 31.45 |
| Solvent         | 42.46 | 50.12 | 45.28 | 40.18 | 40.47 | 50.06 | 29.22 | 34.55 | 20.35 | 28.10 |
| Plasticizer     | 16.68 | 17.72 | 20.82 | 15.07 | 13.36 | –     | 29.61 | 35.65 | 28.28 | 20.05 |
| Addition        | –     | 6.55  | 6.58  | 13.86 | 14.91 | 6.32  | 11.76 | 6.35  | 14.66 | 19.50 |
| No. texture layers | 5     | 5     | 6     | 6     | 7     | 6     | 6     | 6     | 6     |
| Hot pressing at 177°C for 2 hours |          |         |         |         |         |         |         |         |         |         |

Note: The fibers used in the making of the fabric does not present surface treatment

**Figure 1.** Scheme of the experimentation program for sintered polymer composite materials (DYCY-50%; DEDA-12%).

Experimentation program aims to:
- to determine the effect of the high content of graphite composite sintered metal material on the physical-mechanical and tribological characteristics;
- to determine the effect of pressing and sintering process parameters on the physical-mechanical and tribological characteristics of the materials developed. Mechanical and tribological characteristics values specific of the polymer composite materials investigated are presented in Tables 1, 2. Based on the data presented in these tables it was traced a set of graphs for specific tribological evaluation of this type of material. It shows the variation coefficient of friction depending on the strength, the sliding speed constant values (v = 7.2 mm/s) and the normal load N = 8 daN.

**Table 1.** Characteristics determined for composite materials made with resin epoxy curing cold and short carbon fibers.

| No. | Test type | Fiber volume fraction, % | Hardener, % | Resin type | Porosity, % | Expansion coefficient (l/°C) | Mechanical resistance, daN/mm² | Friction coefficient |
|-----|-----------|--------------------------|-------------|------------|-------------|-----------------------------|-------------------------------|---------------------|
| 1.  | TB1       | 27.9                     | DEDA-12     | IV         | 4.02        | 0.87                        | 2.41                          | 0.24                |
| 2.  | TB2       | 20.8                     | DEDA-12     | V          | 12.40       | 0.97                        | 1.92                          | 0.22                |
| 3.  | TB3       | 28.4                     | DEDA-12     | VI         | 19.53       | 1.62                        | 2.28                          | 0.27                |
| 4.  | TB4       | 28.1                     | DEDA-12     | 0          | 6.04        | 0.69                        | 3.55                          | 0.25                |
| 5.  | TB5       | 24.6                     | DEDA-12     | 5          | 12.16       | 3.26                        | 2.48                          | 0.28                |

**Table 2.** Characteristics determined for composite materials made with modified single-component epoxy resin reinforced with carbon fiber.

| No. | Test type | Fiber volume fraction, % | Resin, % | Density (g/cm³) | Porosity % | Expansion coefficient (l/°C) | Mechanical resistance, daN/mm² | Friction coefficient |
|-----|-----------|--------------------------|----------|----------------|------------|-----------------------------|-------------------------------|---------------------|
| 1.  | T1        | 46.7                     | 29.79    | 1.302          | 11.25      | 4.06                        | 0.34                          | 0.26                |
| 2.  | T2        | 38.25                    | 41.66    | 1.401          | 13.15      | 9.747                       | 0.86                          | 0.22                |
| 3.  | T3        | 40.4                     | 30.36    | 0.327          | 16.01      | 4.07                        | 0.6                           | 0.22                |
| 4.  | T4        | 48.71                    | 43.92    | 1.392          | 12.72      | 4.07                        | 0.86                          | 0.21                |
| 5.  | T5        | 53.7                     | 30.32    | 1.728          | 15.05      | 4.19                        | 0.69                          | 0.25                |
| 6.  | T6        | 47.15                    | 29.25    | 1.775          | 11.16      | 9.709                       | 0.41                          | 0.21                |
| 7.  | T7        | 35.43                    | 29.41    | 2.018          | 11.12      | 9.963                       | 2.17                          | 0.19                |
| 8.  | T8        | 46.43                    | 36.71    | 1.920          | 15.25      | 9.565                       | 5.77                          | 0.28                |
| 9.  | T9        | 48.48                    | 36.4     | 1.970          | 14.99      | 10.240                      | 4.52                          | 0.22                |

3. Analysis of composite materials sintered with aluminum matrix reinforced with carbon fibers having different orientation

With this type of composite materials, their behavior on friction and wear was determined by three types of orientations of fibers, the tests were carried out on a standard machine of tribology needle disk type with counter disc made of gray cast iron with a surface hardness of HB 92. It is known that metal material properties such as density, modulus of elasticity and thermal expansion can be substantially changed by their reinforcement with fibers or with particles. It is evident that if matrices reinforced with fibers oriented to the direction of sliding the wear mechanisms will be different for the three extreme cases: normal (N type), parallel (P) and antiparallel-perpendicular (type AP). Regarding the experiments program, were effectuate tests on samples from the Al matrix composites and 41% carbon fiber. The table shown below the properties that have the fiber and matrix, the material being manufactured by bonding technology through diffusion.
Figure 2. Graph of the characteristics determined for composite materials made with modified single-component epoxy resin reinforced with carbon fiber.

The effect that heat treatment had on the micro-hardness matrix and the tensile strength of the composite are shown in Table 3.

| Material | Hardness (kgf/mm²) | Mechanical resistance (MPa) |
|----------|--------------------|-----------------------------|
| On reception | 78.8              | 735                         |
| 450°C, 24 h   | 62.0              | 615                         |
| 500°C, 24 h   | 55.0              | 580                         |
| 545°C, 24 h   | 68.2              | 405                         |
| 545°C, 258 h  | 64.6              | 420                         |

Table 4 shows the average speed values of hardness and wear applied to each sample and each fiber orientation. Also, the effects that heat treatment had on the coefficient of friction heat treatment are presented in Table 5.

| Material | Wear rate [(cm³/cm) x 10⁷] | Hardness (kgf/mm²) |
|----------|-----------------------------|--------------------|
| On reception | 0.0075±0.0001             | 78.8               |
| 545°C, 24 h   | 0.0099±0.0011             | 68.2               |
| 545°C, 168 h  | 0.0123±0.0064             | 64.6               |
| 450°C, 24 h   | 0.0126±0.0034             | 62.0               |
| 500°C, 24 h   | 0.0204±0.0070             | 55.0               |

Table 5. The effects it has on the speed stationary heat treatment of wear test samples submitted with fibers perpendicular to the direction of sliding.

| Material | Wear rate [(cm³/cm) x 10⁷] | Hardness (kgf/mm²) |
|----------|-----------------------------|--------------------|
| On reception | 0.0075±0.0001             | 78.8               |
| 545°C, 24 h   | 0.0099±0.0011             | 68.2               |
| 545°C, 168 h  | 0.0123±0.0064             | 64.6               |
| 450°C, 24 h   | 0.0126±0.0034             | 62.0               |
| 500°C, 24 h   | 0.0204±0.0070             | 55.0               |
Table 6. The effects it has on the heat treatment on the stationary friction coefficient presented on the test samples with normal, parallel and perpendicular fibers to counter friction disk.

| Material          | Friction coefficient | Material          | Friction coefficient | Material          | Friction coefficient |
|-------------------|----------------------|-------------------|----------------------|-------------------|----------------------|
| 450°C, 24 h       | 0.132±0.005          | 450°C, 24 h       | 0.146±0.003          | 450°C, 24 h       | 0.136±0.006          |
| 500°C, 24 h       | 0.142±0.004          | 545°C, 168 h      | 0.149±0.006          | 545°C, 24 h       | 0.137±0.016          |
| 545°C, 24 h       | 0.144±0.004          | On reception      | 0.152±0.003          | 545°C, 24 h       | 0.139±0.006          |
| 545°C, 168 h      | 0.148±0.003          | 545°C, 24 h       | 0.160±0.008          | 500°C, 24 h       | 0.146±0.002          |
| On reception      | 0.148±0.002          | 500°C, 24 h       | 0.167±0.006          | On reception      | 0.175±0.007          |

Based on the data presented in these tables it was traced a set of graphs for specific tribological evaluation of this type of material. It shows the variation coefficient of friction depending on the strength, at the constant values of the sliding speed (v = 7.2 mm/s) and the normal load N = 8 daN for different orientations of fiber.

Table 7. Mechanical and tribological characteristics of the Al matrix composites reinforced with carbon fibers.

| No. | Material       | Mechanical resistance, MPa | Friction coefficient | N | P | AP |
|-----|----------------|---------------------------|----------------------|---|---|---|
| 1.  | Reception state| 735                        | 0.132                | 0.146|0.136|
| 2.  | 450°C, 24h     | 615                        | 0.142                | 0.149|0.137|
| 3.  | 500°C, 24h     | 580                        | 0.144                | 0.152|0.139|
| 4.  | 545°C, 24h     | 405                        | 0.148                | 0.16 |0.146|
| 5.  | 545°C, 24h     | 420                        | 0.148                | 0.167|0.175|

4. Conclusions

Microscopy tests of the surfaces subject to wear on the TB4 specimen showed a stretch tendency of the fibers before their breaking and resin zones have the appearance of the molten area figure 4 loose particles observed in the test areas captured wear and molten resin. These affirmations are also supported by the surface appearance of the specimen before the test wear.
Knowing that for $\mu = 0.08 \div 0.25$ the materials are optimal in terms of anti-friction applications, and for $\mu = 0.25 \div 0.45 (0.55)$ are designed for friction tests, it is noted that other appropriate materials T7 and T3 have a clear anti-friction behavior, while those for tests T5, T1, T8 have a specific behavior of friction materials. Other materials have coefficients of friction with intermediate values, possibly through changes in composition can be classified in one of two areas.

Polymer composite materials reinforced with carbon fiber texture is remarkable that at this sliding rate, most samples have low values for the coefficient of friction. For the normal fibers to the plan and the sliding direction, the wear rate decreases with decreasing of the hardness of matrix. Opposite trend, increasing hardness with decreasing of the wear rate, was observed in materials with parallel fiber to the sliding plane and the sliding direction. If the fibers were parallel to the plane and sliding direction, it was found a clear correlation between the wear rate and thickness of the reaction zone, wear rate increase with increasing of the reaction zone.

Can be observed that are obtained higher values for the friction coefficients of the material in the reception state than the heat-treated material, seeming that fiber orientation has a more pronounced effect on the friction coefficient than heat treatment.

In the wear stationary phase, when the fibers are parallel to the sliding plane, the microscopic investigations showed that the matrix material is removed in layers, leaving most exposed fiber until the frictional forces create a spinning moment to the outwards on the exposed fibers. The tensions created by moment are normal, the brittle interface giving them a little resistance.

Also, when are propagate the cracks meet fiber but instead to pass through the fibers, spread around the fibers from the fragile area of reaction, eliminating layers of matrix. In both cases, variations relatively low in the properties of the interface (resulting from heat treatments) are insignificant to the overall resistance of the fiber to small “running outwards” and propagation of cracking in the reaction zone. Investigation of under-superficial cracks of matrix has enabled to conclude that matrix wearing is made by layering mechanism, based on the nucleation and propagation of cracks below (and parallel to) the surface of wear.

If the fibers were normal in the plane and direction of slip-surface appeared smoother than that observed for perpendicular and parallel type tests, where were insignificant rolling and pulling fibers. N-type samples, sub-superficial matrix cracks were propagated perpendicular to the fibers and were diverted along the interface when they came to the fibers. Stratification matrix of fibers, the fibers lose support, break and are extracted from the matrix.

Factor controlling the wear rate is the rate at which remove layers from the matrix of fibers. Crack propagation speed is proportional to the hardness of the material; Therefore, as shown in table 7, for samples of type N, the speed decreases wear while the matrix hardness.
It has been found thus that the wear of the matrix was produced by layering mechanism for all the directions of the fiber.

In the case of normal and perpendicular orientations of the fibers, the rate of wear increased by increasing the toughness of the matrix. In the case of parallel orientation of the fibers, the rate of wear increased by increasing the thickness of the reaction zone. When the fibers are normal to the plane and direction of sliding of the fiber extraction it was minimal.

When the fibers are parallel to the plane of sliding and perpendicular to the direction of sliding of the fiber was significant running outwards. When the fibers are parallel to the sliding direction, extracting the fiber was significant.

The effect that it had on the rate of attrition fiber orientation was more marked for heat treated materials than for materials in the receiving state.

Could not establish any relationship between hardness but the matrix or the thickness of the reaction zone and friction coefficients; however, the coefficients of friction have minimum values when the fibers are normal to the friction counter disc.

5. References
[1] Kunze, J.M. and Bampton, C.C. 2001 Challenges to Developing and Producing MMCs for Space Applications, Journal of Minerals, Metals & Materials 22-25
[2] Bunsell, A.R. 2005 Oxide Fibers for High-Temperature Reinforcement and Insulation, Journal of Minerals, Metals & Materials 48-51
[3] Davim, J.P. 2000 Comparative study of metal matrix composites machinability with diamond cutting tools, Diamond 2000-11th European Conference, Elsevier Sc, Porto, Portugal, 15-19
[4] Huang, Y. and Langdon, T.G. 2003 The Creep Behavior of Discontinuously Reinforced Metal-Matrix Composites, Journal of Minerals, Metals & Materials 15-20
[5] Swolfs Y., Meerten Y., Hine P., Ward I., Verpoest I., Gorbatikh L. 2015 Introducing ductility in hybrid carbon fibre/self-reinforced composites through control of the damage mechanisms, Composite Structures, Volume 131 259-265
[6] Minus, M. and Kumar S. 2005 The Processing, Properties and Structure of Carbon Fibers, Journal of Minerals, Metals & Materials 52-58
[7] Rawal S. 2001 Metal-Matrix Composites for Space Applications, Journal of Minerals, Metals & Materials 14-17
[8] Gonçalves J., dos Santos J.F., Canto L.B., Amancio-Filho S.T. 2015 Friction spot welding of carbon fiber-reinforced polyamide 66 laminate, Materials Letters, Volume 159 506-509
[9] Klotz S., Zanger F., Schulze V. 2014 Influence of Clamping Systems during Milling of Carbon Fiber Reinforced Composites, Procedia CIRP, Volume 24 38-43
[10] Zhang J., Liu S., Lu Y., Yin X., Zhang Y., Li T. 2016 Liquid rolling of woven carbon fibers reinforced Al5083-matrix composites, Materials & Design Vol. 95 89–96
[11] Rittner, M.N. 2000 Expanding World Markets for MMCs, Journal of Minerals, Metals & Materials, November 43