

Article

Basalt Short Fibres Dispersion and Fabric Impregnation with Magnesium Alloy (AZ63): First Results

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Abstract: Magnesium is one of the lightest structural metal used in different industrial sector and many works are present in literature about the study of its reinforcement by fillers addition. Basalt fibres are natural fillers with good mechanical properties, excellent resistance to high temperature and lower cost than carbon fibres. For these aspects, in the last years they are increasingly used in the production of composite materials with polymeric matrices. However, very few information are presents in literature about the use of basalt fibres as reinforcement in metal matrix composite materials. It is well known that the impregnation of fibres reinforcement affects the mechanical behavior of composites materials. The aim of this study is to investigate the impregnation and the behavior of basalt fibres in a magnesium alloy composite material manufactured by a centrifugal casting technique.

Keywords: basalt fibre; fabric; magnesium; centrifugal cast; metal matrix composite

1. Introduction

Metal matrix composite materials (MMC) represent a satisfactory solution to obtain lighter materials with improvement in the typical properties of the metal. Despite this advantage, the production of MMC still presents some issues in terms of difficulty to obtain homogeneous dispersion of reinforcement in the matrix and good impregnation of fillers that affect the mechanical properties of the final composite.

In order to obtain a good interface, it is necessary to evaluate the wettability, that is the ability of a liquid material to distribute uniformly on a solid surface. The wettability between the matrix in the molten state and the reinforcing material depends on several elements, including the intrinsic properties of the material, such as the surface energy of the matrix and of the reinforcement, and the characteristics of the surface of the reinforcement, such as the amount of oxidation and contamination. If is necessary, wettability can be improved by increasing the surface energy of the solid, decreasing the surface tension of the molten metal or decreasing the energy of the particle-matrix interface through the coating of the particles [1][2], adding additives to the melt or subjecting it to ultrasonic irradiation [3]. The use of coupling agents improves the wettability of the fibres and promotes the formation of bonds to the fibre / matrix interface contributing for example to the formation of non-metallic compounds which, if in thin layers, improve wettability but can damage the surface of the fibre. It has been shown that, depending on the interfacial energies, the metal matrix can incorporate or reject the reinforcement during solidification [4]. Another problem in the compatibility of the two phases consists in the temperature at which the molten metal meets the fibres. If too high, in fact, it damages the fibres themselves with consequent repercussions on their mechanical properties. These are the main problems faced in the production of MMCs compared to that of monolithic metallic materials.

Carbon fibres (micro and nano sized) and glass fibres are widely studied in the production of polymeric and metallic composite materials thanks to its physical properties (electrical conductivity

in the case of carbon nanotubes) and mechanical properties (elastic modulus and tensile strength in the case of microfibrils[5] [6]).

Basalt fibres are natural fillers with lower cost than carbon fibres[7], very good resistance to high temperature, compared with glass fibres, and good mechanical properties [8]. The density of about 2.8 g/cm³ is very close to that of glass fibers, although basalt fibers are more resistant [9] and can be used over a wider temperature range, from about -200 ° C to about 800 ° C, while type E glass goes from -60 ° C to 450 ° C [7]. Other advantages of basalt fibers are resistance to alkaline environments, as they can withstand up to pH = 13 - 14, and a strong resistance to the action of fungi and microorganisms [9]. When in contact with other substances, the basalt fibers do not produce chemical reactions that could damage the health or the environment, they are incombustible and resistant to explosions. Furthermore, since chemical additives and / or solvents are not added during production, they are 100% natural and sustainable and their recycling is much more efficient than that of glass fibers. When the resin composites containing basalt are recycled, this is again obtained in the form of powder because, being its melting point decidedly high (1400 ° C), it is the only product that is found after incineration [10]. Typical applications of basalt concern protective and wear-resistant coatings in tanks, pipes and pipelines. The basalt composite pipes, in fact, can be used for the transport of petroleum products and aggressive liquids and for the supply of hot and cold water [11]. Furthermore, they are ideal for fire protection, as fireproof materials in nuclear power plants, and for electrical insulation applications, for example in printed circuits.

despite this, few literature informations are available about the production of basalt fibres reinforced MMC.

In this work, we focused on the production of composite materials with magnesium alloy matrix and basalt fibres reinforcements by induction melting [12] and centrifugal casting.

In particular, the aim of this study is to evaluate the wettability of basalt fibres by the magnesium alloy and compare the behaviour of basalt micro fibres (BMFs) and basalt woven fabric (BWF) in the magnesium alloy matrix produced by the manufacturing process previously mentioned.

2. Materials and Methods

One of the typical magnesium foundry alloys was selected as matrix for the composite manufacturing: AZ63 alloy shavings, containing 6-7 wt% of aluminium and 3-4 wt% zinc. Shavings were provided by COMETOX (Italy) and used without further purifications.

Milled recycle BMFs, with 300 µm of medium length and 18 µm of medium diameter, produced by BASALTEX were selected as short fillers. This type of basalt fibres was used as received. The actual fibre diameters were measured by optical microscopy (Figure 1).

A BWF produced by BASALTEX (Belgium), with a specific surface weight of 220 g/m² and silane sizing agent designed to be compatible with epoxy and thermoplastic resin matrix, was used as long filler. This type of basalt fibres has a nominal diameter of 13 µm and the actual fibre diameters were checked by optical microscopy.

Thermal treatment allows to degrade the sizing on the fibres [8] and in this work were manufactured composites samples using BWF after thermal treatment in air at 400° C for 4 h to avoid problems in terms of adhesion and bubbles production by the sizing at melting temperature of magnesium alloy.

Magnesium composites with short fillers, were prepared by mixing the metal shavings with different wt% of BMFs in a mortar using acetone to facilitate the dispersion of fillers in the metallic shavings. This mixture was charged in a melting pot and prepared to be melted and cast (Figure 2).

Magnesium composites with long fillers were manufactured charging only shavings in the melting pot, while the BWF were positioned and fixed directly in the mould cavity section in a longitudinal position respect to the casting axis (Figure 1). This configuration has allowed to obtain an impregnation of basalt fabrics by the melted magnesium alloy.

In both cases, melting pots (mod. C20) and a graphite mould with a parallelepiped cavity (10 x 5 x 50 mm) produced by F.lli Fossati (Italy) were used.

Both kinds of samples were produced by a Neutor Digital F.lli Manfredi (Italy) induction furnace, that is characterized by the possibility to melt under controlled atmosphere (Ar) and to cast with centrifugal force (Figure 2).

To evaluate the influence of reinforcement, in the case of short fibres, two different percentages of fillers were adopted (5 and 10 wt.%), while, for composites with long fillers, 1 layer and two layers of BWF (\approx 5 and 10 wt.%) were inserted in the composites.

Figure 3 and Figure 4 shows the raw samples obtained opening the die respectively after mixing and casting of composites reinforced with BMFs and after impregnation of BWF.

The samples obtained were cut longitudinal to the casting axe, and perpendicular to the woven fabric. The rods obtained (4 x 3 x 50 mm) and (4 x 2 x 50 mm) respectively for short and long fibres were prepared to the four bending tests [13][14] (at room temperature) with a Zwick Roell Z2.5 testing machine. Before mechanical tests, the samples described were used for evaluation of porosity by density measurement and through ME54 Mettler-Toledo (Italy) analytical balance, equipped with the tool for solid density measurements by the Archimedes principle.

The residual porosity in the composite was calculated with the comparison between the theoretical and measured density of the samples.

The theoretical density was calculated with the rule of mixtures: Composite samples experimental density values were compared with theoretical ones calculated according to the rule of mixtures.

$$\rho_C = \rho_f V_f + \rho_m V_m \quad (1)$$

Where ρ_C is composites density, ρ_f is fibres density, ρ_m is matrix density, V_f is volumetric ratio between fibres and composite and V_m is volumetric ratio between matrix and composite.

Cutting surface and fracture surface were analysed with optical and SEM microscopy (Figure 3 and Figure 4), performed with a NIKON – L150 and a PHILIPS XL-40, (FEI B.V, Eindhoven, The Netherlands) respectively in order to evaluate the dispersion and the impregnation of fillers.

3. Results

Optical microscopy analysis has shown the presence and the well dispersion of the BMFs in AZ63 specimens. In Figure 3 is reported, by way of example, the image of the sample at 5 wt.%, after acid attack with HCl concentrated, to make visible the fibres. SEM analysis on fracture surfaces confirmed the optical analysis results, moreover, Figure 3 shows that the BMFs are well wetted by the metal matrix and partially aligned in the direction of the casting axis, but more defectiveness and porosity are visible on the fracture surface. The presence of residual porosity is confirmed also by the results obtained with the comparison between theoretical and measured density. In the diagram in Figure 6 is possible to see that the composites reinforced with the 5% and 10% of BMFs shows respectively a residual porosity of 2,28% and 1,53%. These values are comparable with the residual porosity measured in the samples without basalt reinforcement and therefore intrinsic to the chosen manufacturing process. Comparing the result of SEM analysis reported in figure 3 and figure 4 is possible to see the different morphology of the fibres in the fractural surface. The composites reinforced with BMFs (Figure 3) shows a higher degradation of the fibres than the ones of the composite reinforced with BWF. This effect it is possible to attribute to the longer residence time of the short fibres at high temperature [15], being mixed with the metal directly in the melting crucible, compared to the fabrics which are impregnated by the liquid metal in a few seconds.

Optical and SEM analysis on BWF samples has shown a well impregnation of fibres by metal matrix but not homogenously throughout the sample's length, in Figure 5 is reported one of the many parts of the BWF not completely impregnated by the metal of the matrix. In Figure 4 on the contrary, is reported the cutting surface of the single layer composite were the metal was completely penetrating between the threads of the fabric.

Figure 4 shows a fracture surface and in it is possible to observe the clear fracture of the basalt fibres with the complete absence of pullout phenomena, indication of an excellent matrix-fibre

interface with a complete transfer of the applied load from the matrix to the reinforcements in these parts of the composite. In this picture are visible also different longitudinal fractures on the fibres probably due to thermal shock caused by the casting of molten metal.

Figure 6 shows a residual porosity in the samples with 5 wt.% and 10 wt.% of BWF respectively of 4,06% and 2,40%.

Mechanical characterization was performed on the two type of composites manufactured and the Young's modulus, in the case of BMFs, increase with the amount of fillers concentration (Figure 7). Flexural strength increases for 5 wt.% of BMFs but decreased with the amount of fillers concentration, because of the amount of defectiveness in the matrix due probably to the effect of the addition of BMFs on the rheological property of the mix pushed in the die only with the centrifugal force.

Mechanical tests on composites with BWF have shown a slight decreasing of Young's modulus but a drastically decreasing of flexural strength, probably due to the embrittlement by thermal shock of the fibres and to the not homogeneously impregnation of fabric (Figure 8).

4. Discussion

In this work the interaction between a typical magnesium alloy (AZ63), and basalt fibres in two different configurations was studied, short fibres and woven fabric, in order to evaluate the possibility to produce MMC materials with a particular manufacturing process based on the induction melting and centrifugal casting.

Result of SEM observation crossed with the density measurement performed on the composites reinforced with BWF confirms the presence of higher residual porosity in this type of composites than the AZ63 samples and the composites obtained with BMFs reinforcement.

Despite the difficulty and the residual porosity measured in the sample, the results show a good interfacial interaction between the matrix and the fillers, in particular in the case of composites with the addition of 5 wt.% of BMFs; in fact, at the expense of a weight increase of only 0,92% it was registered an increase of Young's modulus of around 20% and an increase of 10% in terms of flexural strength.

From the results obtained, basalt fibres can be considered a good reinforcement for magnesium alloys thanks to the good wettability and strong interfacial interaction between them.

Centrifugal casting manufacturing process needs some optimization in terms of operative parameter principally to eliminate the residual porosity, detected in the samples produced, and to obtain an optimal impregnation of the BWF.

Preheating the mould with the fabric inside and varying the rotation speed of centrifugal casting system are two of the possible strategies to optimize the manufacturing process respectively to avoid the effect of thermal shock and to ensure a good fluidity of molten metal during the casting process.

Appendix B

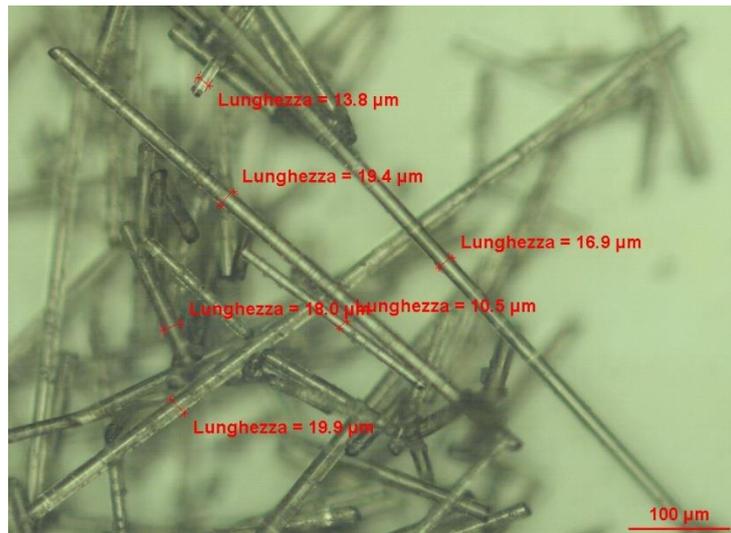


Figure A1: Milled recycle BMFs

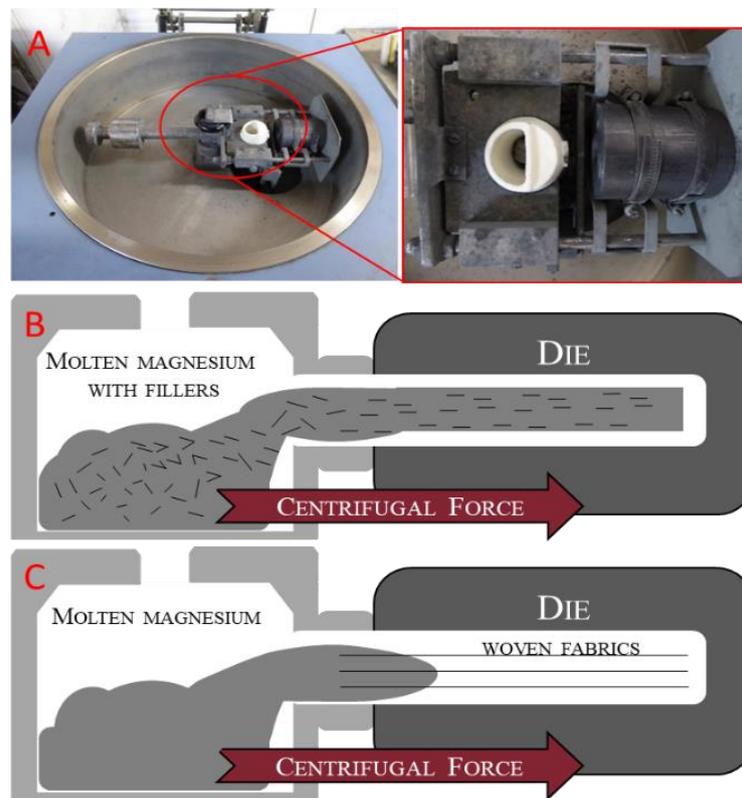


Figure A2 – A) Induction melting and centrifugal casting furnace – B) Schematization of method used with short basalt fibres – C) Schematization of method used with Basalt woven fabric

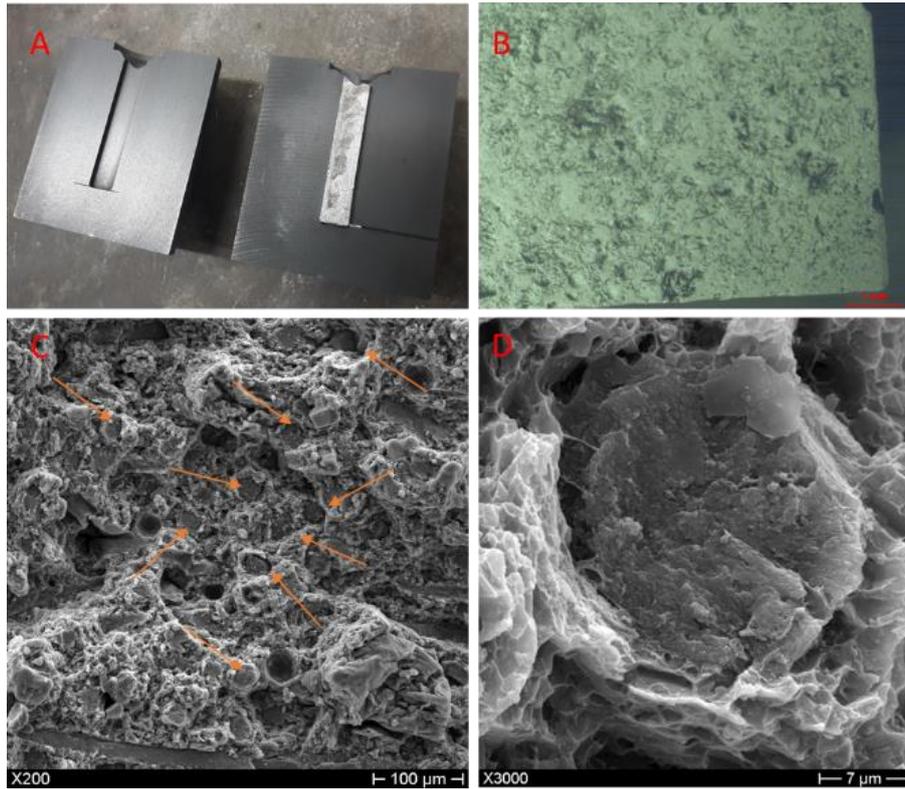


Figure A3 - A) Sample after cast with short fillers; B) Cutting surface after acid treatment; C) Fracture surface of sample with short fillers; D) Enlargement on one single short basalt fibre.

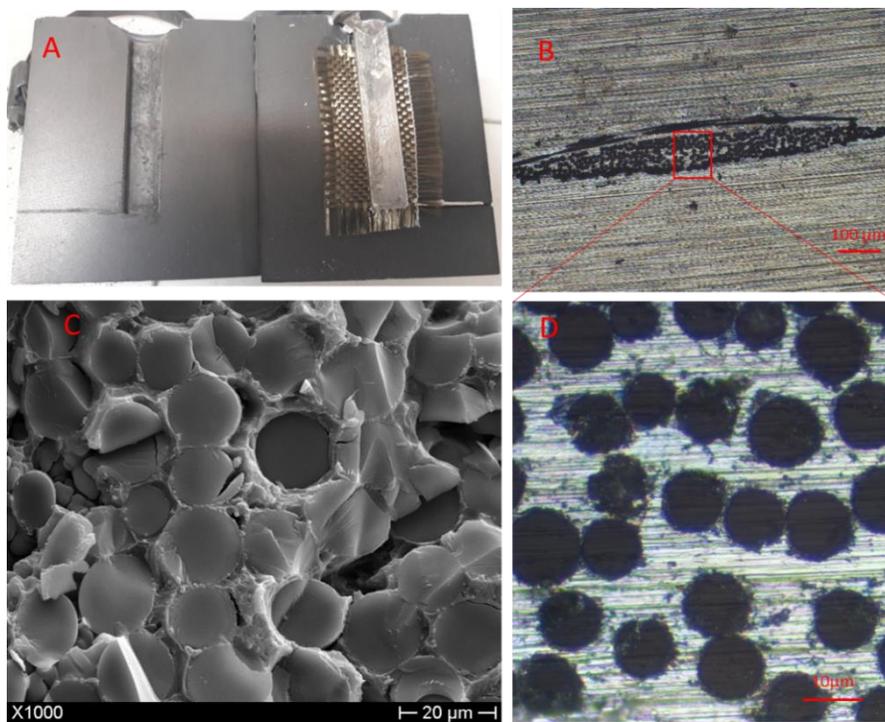


Figure A4 - A) Sample after cast with long fillers; B) Cutting surface of sample with long fillers; C) Fracture surface of sample with long fillers; D) Enlargement of Cutting surface in B.

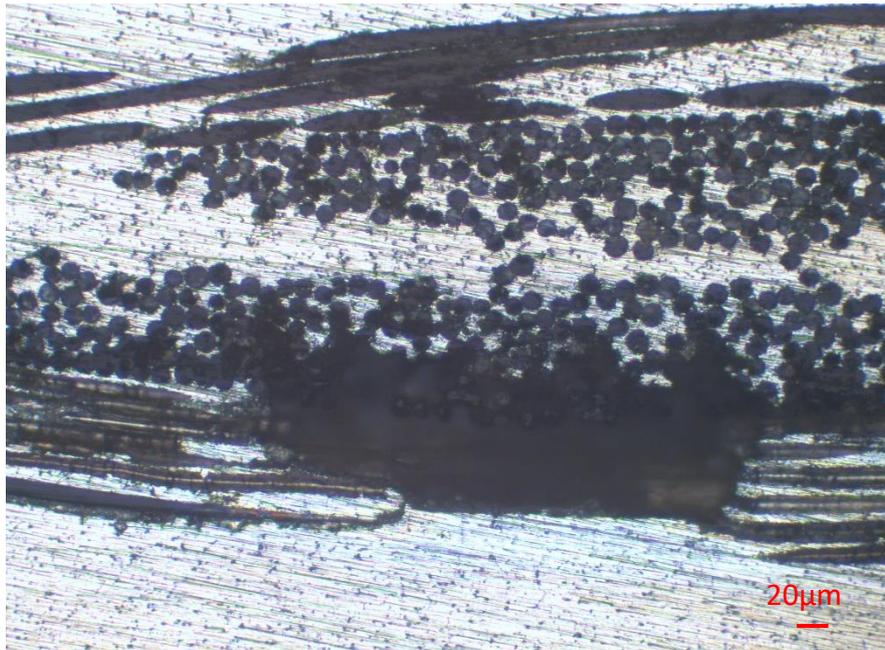


Figure A5- Not homogenous impregnation of BWF by molten AZ63

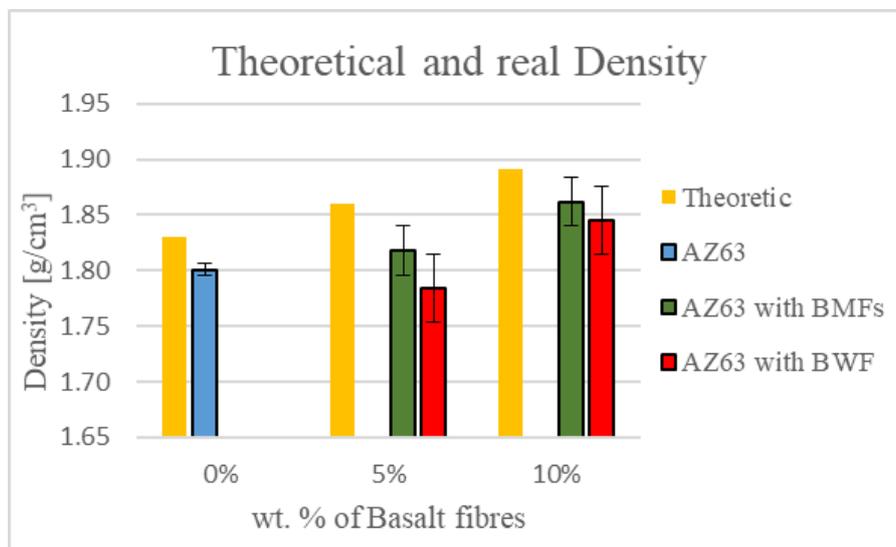


Figure A6- Comparison of theoretical and measured density of composites obtained.

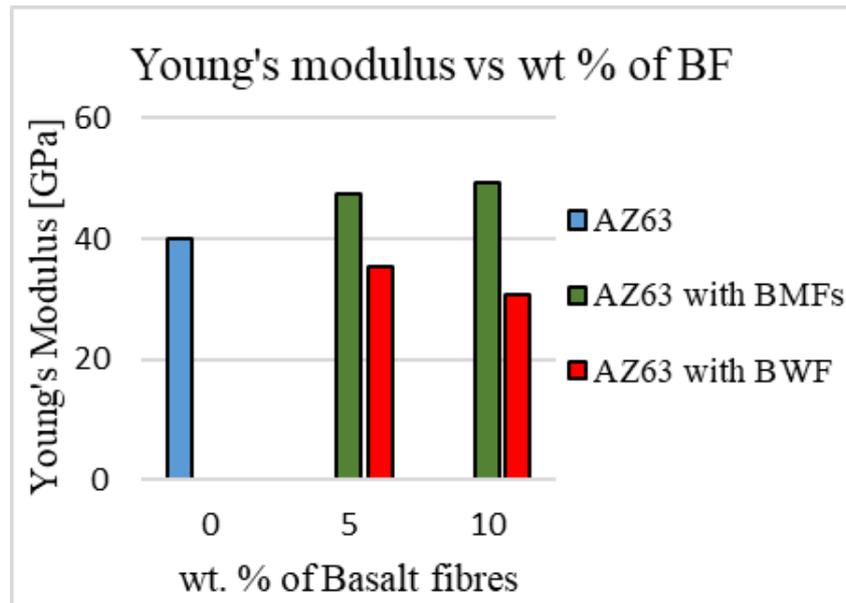


Figure A7 - Young's modulus for the two kinds of samples.

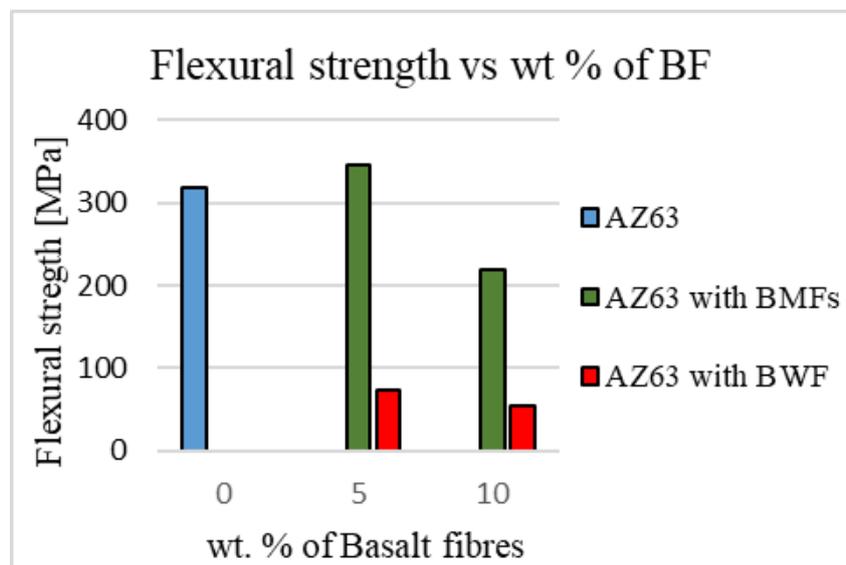


Figure A8 - Flexural strength trend for the two kinds of samples.

References

1. M. Valente, D. Marini, V. Genova, A. Quitadamo, F. Marra, and G. Pulci, "Lightweight metallic matrix composites: Development of new composites material reinforced with carbon structures," *J. Appl. Biomater. Funct. Mater.*, vol. 17, no. 1_suppl, p. 228080001984029, 2019.
2. V. Genova, D. Marini, M. Valente, F. Marra, and G. Pulci, "Nanostructured Nickel Film Deposition on Carbon Fibers for Improving Reinforcement-matrix Interface in Metal Matrix Composites," vol. 60, pp. 73–78, 2017.
3. H. Z. Ye and X. Y. Liu, "Review of recent studies in magnesium matrix composites,"

- J. Mater. Sci.*, vol. 39, no. 20, pp. 6153–6171, 2004.
4. J. Hashim, L. Looney, and M. S. J. Hashmi, “Particle distribution in cast metal matrix composites - Part I,” *J. Mater. Process. Technol.*, vol. 123, no. 2, pp. 251–257, 2002.
 5. D. Marini, V. Genova, F. Marra, G. Pulci, and M. Valente, “Mechanical Behaviour with Temperatures of Aluminum Matrix Composites with CNTs,” vol. 60, pp. 25–30, 2017.
 6. X. Wei, L. Qi, L. Ju, W. Tian, X. Hou, and H. Li, “Effect of holding pressure on densification and mechanical properties of Cf/Mg composites,” *Mater. Sci. Technol. (United Kingdom)*, vol. 33, no. 5, 2017.
 7. P. Kiekens and V. L. L., “Basalt fibres as reinforcement for composites,” pp. 5–6.
 8. M. F. Pucci, M. C. Seghini, P. J. Liotier, F. Sarasini, J. Tirilló, and S. Drapier, “Surface characterisation and wetting properties of single basalt fibres,” *Compos. Part B Eng.*, vol. 109, pp. 72–81, 2017.
 9. V. Dhand, G. Mittal, K. Y. Rhee, S. J. Park, and D. Hui, “A short review on basalt fiber reinforced polymer composites,” *Compos. Part B Eng.*, vol. 73, pp. 166–180, 2015.
 10. T. Bhat, D. Fortomaris, E. Kandare, and A. P. Mouritz, “Properties of thermally recycled basalt fibres and basalt fibre composites,” *J. Mater. Sci.*, vol. 53, no. 3, pp. 1933–1944, 2018.
 11. H. Jamshaid and R. Mishra, “A green material from rock: basalt fiber – a review,” *J. Text. Inst.*, vol. 107, no. 7, pp. 923–937, 2016.
 12. M. Mansoor and M. Shahid, “Carbon nanotube-reinforced aluminum composite produced by induction melting,” *J. Appl. Res. Technol.*, 2016.
 13. G. Pulci, M. Tului, J. Tirillò, F. Marra, S. Lionetti, and T. Valente, “High temperature mechanical behavior of UHTC coatings for thermal protection of re-entry vehicles,” *J. Therm. Spray Technol.*, vol. 20, no. 1–2, pp. 139–144, 2011.
 14. L. Baiamonte *et al.*, “Tribological and high-temperature mechanical characterization of cold sprayed and PTA-deposited Stellite coatings,” *Surf. Coatings Technol.*, vol. 371, no. August 2018, pp. 322–332, 2019.
 15. J. Militký, V. Kovačič, and J. Rubnerová, “Influence of thermal treatment on tensile failure of basalt fibers,” *Eng. Fract. Mech.*, vol. 69, no. 9, pp. 1025–1033, 2002.