Tribological Behavior of 316L Stainless Steel Reinforced with CuCoBe + Diamond Composites by Laser Sintering and Hot Pressing: a Comparative Statistical Study

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Abstract. The aim of this work was to assess how the tribological properties of a laser textured 316L stainless steel reinforced with CuCoBe - diamond composites are affected by diamond particles size, type of technology (laser sintering and hot pressing) and time of tribological test. A statistical analysis using IBM® SPSS software was performed. After describing the response variables, the Friedman's test was used to compare how the coefficient of friction varied among samples in five-time points. From this test, results showed that there was no statistically significant difference in the coefficient of friction mean values over the selected time points. Then, the two-samples K-S test was used to test the effect of the diamond particles size and the type of technology on the mean of COF over time. The results showed that, for both sintering techniques, the size of the diamond particles significantly affected the values of the coefficient of friction (p-value < 0.05), whereas no statistical differences were found between the tested sintering techniques (p-value > 0.05). Also, the two-way ANOVA test was used to evaluate how these factors influence the specific wear rate, which conducted to the same conclusions drawn for the previous test. Therefore, it was concluded that the coefficient of friction and the specific wear rate were statistically affected by the diamond particles size, but not by the sintering techniques used in this work.

Keywords: Statistical analysis, Tribological behavior, Laser sintering, Hot Pressing, Multi-material surface.

1 Introduction

Austenitic stainless steels are characterized by high applicability in the mechanical, chemical and process industries [1] due to their high resistance to corrosion, high strength and machinability [2]–[4]. However, the 316L stainless steel (SS316L), in particular, has low wear resistance, which can be improved by the addition of hard ceramic particles on the surface [5]–[7]. These metal matrix composites (MMCs) are increasingly used in industries that require high mechanical and tribological properties [8], [9] because they permit an excellent combination of properties and performance [10], [11].

The 316L stainless steel is a material that presents a chemical composition similar to material used in the fabrication of piston rings [12]. When compared to ductile or cast iron, its inherent strength creates less chance of ring breakage, with consequently longer service life. However, the reduction of the friction, the retention of oil during operation, the retention of particles from wear and the increase in the thermal conductivity are some of the target properties in the development of automotive piston rings [13], [14]. Therefore, the surface of a piston ring must be multifunctional. Some coatings have already been tried at the surface of this mechanical component in order to improve the surface properties [15].

Selective Laser Sintering (SLS) is an additive manufacturing (AM) process that presents an extraordinary versatility to geometry and materials design, as it is considered a near-net-shape technology [16], [17]. It is also characterized by being a fast technique, which is a potential advantage in mass production. On the other hand, Hot Pressing technique (HP), characterized by the simultaneous application of temperature and pressure, allows obtaining well-consolidated components with high mechanical performance due to the low porosity that it presents [18], [19]. When using pressure and temperature simultaneously, it is possible to compensate temperature with pressure, using lower temperatures. In this case, it is important because graphitization of the diamond occurs at 900 °C [20]–[22].

The present work focuses on the development of a multi-functional/multi-material surface with specific areas for specific functions. In this particular case, part of the surface will be prepared to wear behaviour function while the remaining area is still available for other functions mentioned. The influence of two consolidation technologies (laser sintering and hot pressing) and different reinforcement sizes on the sintering of diamond-reinforced composite materials on 316L stainless steel samples will be discussed in this work.

2　　Methodology procedure

2.1　　Materials and processing details

Samples of SS316L with 14 mm diameter were textured through an Nd: YAG laser (Sisma Laser) with a wavelength of 1064 nm, laser power of 6 W, scan speed of 128 mm/s, number of passes of 16 and fill spacing of 5 μm (see Fig. 1).
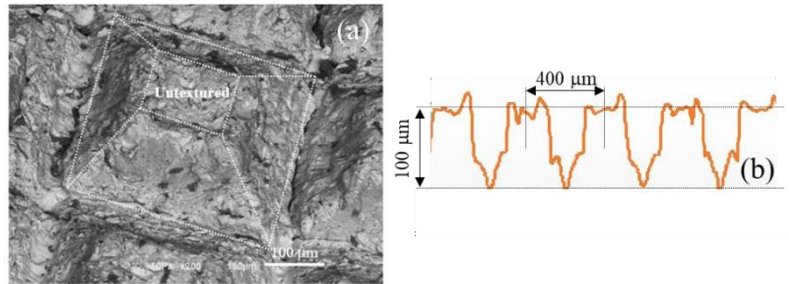

Fig. 1. Texture produced: (a) SEM image of the textured and (b) heights of the profile produced.

The CuCoBe (1.5 wt% cobalt, 0.5 wt% beryllium and the remainder is copper) + 5.8 wt.% diamond composites were produced by mechanical alloying (MA) from elemental powders. Two different grades of diamond particles (0.1-0.5 μm and 40-60 μm) were used. The powder size of the CuCoBe alloy was 40-80 μm. Five different samples were produced by laser sintering and hot pressing in this work (Table 1).

Table 1. Samples produced and analyzed in this work.

Sample reference	Description
316L + C_0.1-0.5 (LS)	Textured 316L stainless steel reinforced with mechanical alloyed CuCoBe + diamond particles (0.1-0.5 μm) produced by LS
316L + C_40-60 (LS)	Textured 316L stainless steel reinforced with mechanical alloyed CuCoBe + diamond particles (40-60 μm) produced by LS
316L + C_0.1-0.5 (HP)	Textured 316L stainless steel reinforced with mechanical alloyed CuCoBe + diamond particles (0.1-0.5 μm) produced by HP
316L + C_40-60 (HP)	Textured 316L stainless steel reinforced with mechanical alloyed CuCoBe + diamond particles (40-60 μm) produced by HP

Fig. 2 shows a schematic representation of the two processes used for the reinforcement of the textured 316L stainless steel, SLS and HP.
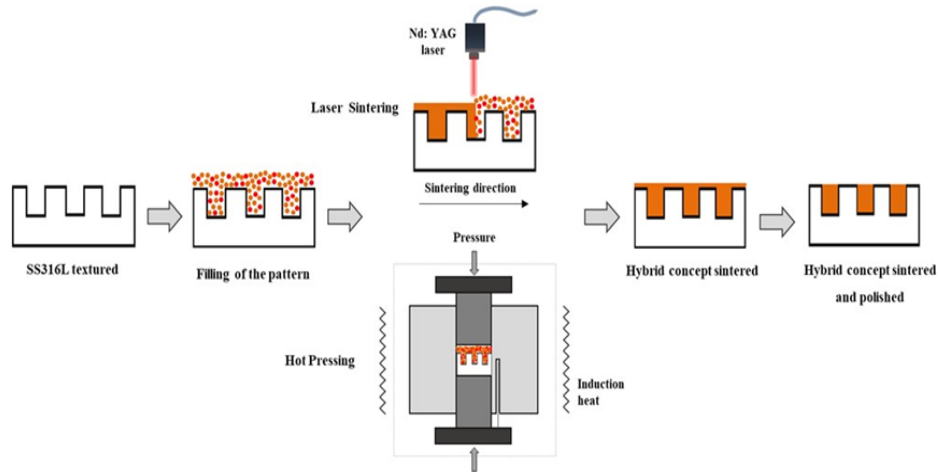
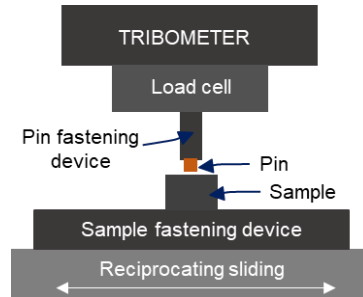**Fig. 2.** Schematic representation of the two processes used for the reinforcement of the textured 316L.

For SLS, samples were sintered through the same laser, power and scan speed used for texturing but using just 1 pass and a fill spacing of 20 μm. Under these conditions one line affects a distance of 27 μm, so with the fill spacing used, overlapping of the lines was ensured and consequently sintering of the entire surface was performed. Regarding HP, samples were heated up to 900 ºC, with a heating rate of 100 ºC/min, and an applied pressure of 70 MPa, for 30 min. The samples were then cooled down to the room temperature.

After sintering, a polishing operation was necessary to expose the 316L steel surface. The samples were polished with SiC abrasive papers down to a 4000 mesh and ultrasonically cleaned with isopropyl alcohol before tribological tests.

2.2 Tribological tests

A reciprocating pin-on-plate tribometer (see Fig. 3) was used (Plint TE67-HT) for the tests, which replicates the piston ring-cylinder liner contact. The pin (counter body) consisted of a malleable cast iron surface, with a geometry that was similar to the engine cylinder body.

The test conditions were defined based on the engine's operating conditions (similar to the in-service conditions) and in accordance with the restrictions of the test equipment. The wear sliding tests were performed dry at 25 N loading (nominal), with a frequency of 1.5 Hz and 7 mm of total stroke length for 4h. Three tests were performed for each sample.

Fig. 3. Schematic representation of the tribological tests.

From the tests it was possible to obtain the COF directly and, in addition, the mass loss of the sample was determined (difference between the initial and the final mass). The mass loss and the density of the materials allowed to determine the wear volume (w) in mm^3. So, the specific wear rate (k) of the surfaces was calculated according to the equation 1.

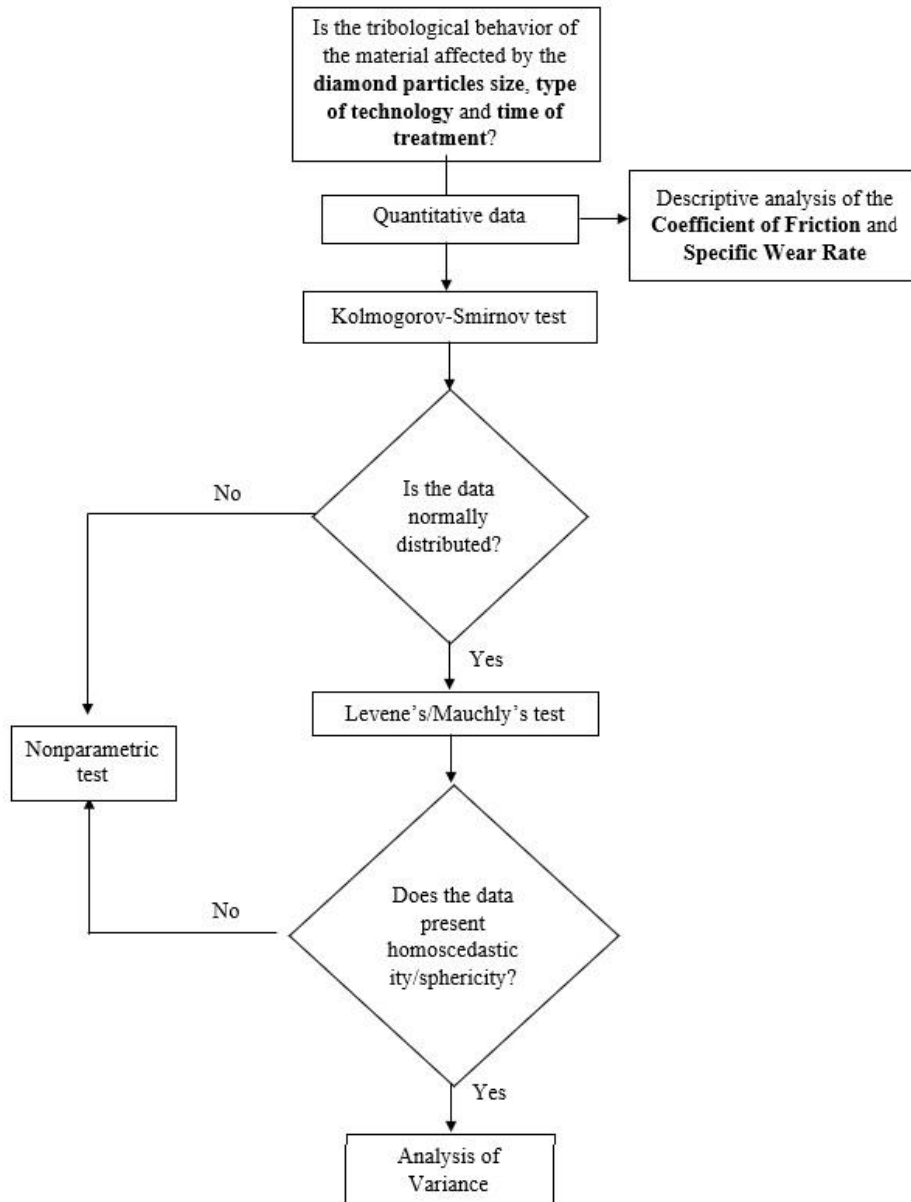$$k = w/(Fn.s) \tag{1}$$

where F_n represents de normal force in N (25 N) and s is the sliding distance in m ($\cong$ 284 m).

It should be noted that while the COF variable is measured over time, the variable specific wear rate is a unique value, measured at the end of each test.

2.3 Statistical Analysis

The general procedure followed in this statistical study is schematically presented in Fig. 4.

Fig. 4. Flowchart of the methodology adopted for the statistical analysis.

3   Statistical analysis and discussion of results

3.1   Descriptive analysis of COF

In this section, a statistical study of the dependent variable, Coefficient of Friction (COF), in each sample [316L + C_0.1-0.5 (LS), 316L + C_40-60 (LS), 316L + C_0.1-0.5 (HP) and 316L + C_40-60 (HP)] is presented considering each trial. It was carried out in SPSS software. Three trials per sample were executed. The obtained results are presented in Table 2.

The descriptive statistics of each sample considering the three trials performed is subjected to discussion. An example is given for Trial 2 of 316L + C_0.1-0.5 (LS). The same analysis is applicable to Trials 1 and 3, as well as to other samples. Trial 2 presents a mean and median of 0.4327 and 0.4324, respectively. This leads to the conclusion that, for Trial 2, the mean COF value is 0.4327 and half the COF values are less than or equal to 0.4324.

For this analysis, the following assumptions were made regarding to the skewness statistic:

Table 2. Statistics for each sample considering each trial for COF.

Material \ Statistics		Mean	Median	Std. Deviation	Skewness
316L + C_0.1-0.5 (LS)	Trial 1	0.4690	0.4637	0.2112	0.2600
	Trial 2	0.4327	0.4324	0.0087	0.1040
	Trial 3	0.4484	0.4574	0.02416	-0.7920
316L + C_40-60 (LS)	Trial 1	0.1195	0.1192	0.0029	0.3620
	Trial 2	0.1309	0.1305	0.0033	0.3000
	Trial 3	0.1320	0.1326	0.0035	0.2120
316L + C_0.1-0.5 (HP)	Trial 1	0.5837	0.5939	0.0460	-0.3820
	Trial 2	0.5446	0.5313	0.0286	0.2970
	Trial 3	0.4926	0.4930	0.0115	-0.3280
316L + C_40-60 (HP)	Trial 1	0.1383	0.1375	0.0039	0.6160
	Trial 2	0.1572	0.1567	0.0034	0.5350
	Trial 3	0.1158	0.1145	0.0064	0.8220

According to the previous assumptions, COF's skewness for Trial 2 (0.1040) presents an approximately symmetric distribution.

Comparing all histograms from the samples, it is possible to conclude that Trial 1 is the worst regarding to its distribution. This might have due to some aspects of the experiments performed. The same counter body was utilized for all trials. In Trial 3, the counter body has a smoother surface with fewer asperities than in Trials 1 and 2, and fewer rough edges are encountered. Therefore, the contact between the two materials (counter body and sample) becomes more uniform. Due to this fact, the relative movement is more easily maintained when compared to Trials 1 and 2. In Trial

1, the surfaces of the counter body and sample are more irregular at a microscopic level (larger number and size of asperities) and therefore there is higher resistance. This leads to higher difficulty in sliding and consequently, a distribution for COF that is not close to normality. Over time and trials, less rough edges exist, and the track adapts to the counter body geometry, leading to better distribution for COF in Trial 3.

3.2 Descriptive analysis of specific wear rate

In this section a statistical study of the dependent variable, specific wear rate (k), for each sample [316L + C_0.1-0.5 (LS), 316L + C_40-60 (LS), 316L + C_0.1-0.5 (HP) and 316L + C_40-60 (HP)] is presented. The analysis was carried out in SPSS software, and the obtained results are presented in Table 3.

Table 3. Statistics for each sample considering each trial for k.

Statistics Sample	Mean	Median	Std. Deviation	Skewness
316L + C_0.1-0.5 (LS)	0.00002733	0.00002410	0.00000862	1.451
316L + C_40-60 (LS)	0.00000236	0.00000265	0.00000223	-0.583
316L + C_0.1-0.5 (HP)	0.00001180	0.00001200	0.00000111	-0.782
316L + C_40-60 (HP)	0.00000058	0.00000109	0.00000148	-1.356

From the results obtained, it is possible to conclude that the 316L + C_0.1-0.5 (LS) sample has the higher mean value of the specific wear rate. Contrarily, the 316L + C_40-60 (HP) sample is the one presenting the lower specific wear rate. Considering the effect of the particle size, these results show that the samples with higher particles sizes are more resistant to wear than samples with lower particles sizes. Regarding the median value, the 316L + C_40-60 (HP) sample experienced the lowest value for this statistic, meaning that 50 % of this sample is subjected to less wear than 50% of the other samples during the performed trials. These results corroborate the above-mentioned conclusion on the mean value for k.

Regarding the skewness statistic for the analysis of k, only one sample [316L + C_0.1-0.5 (LS)] has a right skewed distribution, with the remaining samples comprising left skewness. Taking the above assumptions in consideration, it can be said that the distribution of 316L + C_0.1-0.5 (LS) is highly skewed to the right, that is, it's right tail is longer than the left tail, and the k distribution is more concentrated on the left side. This is corroborated by the fact that the mean k value for this sample is higher than the median value, which in turn is higher than the mode (0.00002733 > 0.00002410 > 0.00002080). Amongst samples comprising a left skewed distribution and considering the previously referred assumptions, the 316L + C_40-60 (LS) and 316L + C_0.1-0.5 (HP) ones present a moderately left skewed distribution, and the 316L + C_40-60 (HP) one has a highly left skewed distribution.

3.3 Differences of the measurement of COF through time

Problem. In order to assess if there is a statistically significance difference of COF values of the samples with respect to time, 5 different time points were selected among the time range. Table 4 presents the five different time points, and the COF values for the four different material conditions, each with three samples, resulting in a total of 12 different conditions.

Table 4. COF values of the 4 different samples at the 5 time points.

Time (s) Sample	3600	6300	9000	11700	14397
316L + C_0.1-0.5 (LS)	0.4473 0.4389 0.4109	0.4423 0.4294 0.4252	0.4621 0.4389 0.4463	0.4837 0.4384 0.4755	0.5058 0.4351 0.4654
316L + C_40-60 (LS)	0.1255 0.1383 0.1389	0.1215 0.1383 0.1330	0.1192 0.1300 0.1330	0.1165 0.1297 0.1280	0.1162 0.1250 0.1270
316L + C_0.1-0.5 (HP)	0.4980 0.5144 0.4888	0.5643 0.5142 0.4806	0.5919 0.5262 0.4905	0.6355 0.5825 0.5034	0.6406 0.5928 0.4847
316L + C_40-60 (HP)	0.1484 0.1629 0.1288	0.1408 0.1607 0.1187	0.1344 0.1564 0.1113	0.1383 0.1579 0.1087	0.1379 0.1556 0.1187

Resolution. In order to study if there is an influence of time in the response variable (COF of the 4 different samples), the first method attempted to use was the repeated measures ANOVA. This method enables to eliminate sources of variability between subjects (samples) on the experiment error [23]. Therefore, the null hypothesis (H_0) for this test, is given as follows:

H_0: The COF mean value of the 12 different subjects is the same at all the 5 time points.

The method implies that the response variable has to be normally distributed within each time points, homoscedasticity and sphericity. Considering the Mauchly's test, $\chi 2$ (4) = 2.760, p-value = 8.66e-09 (< 0.05), the assumption of sphericity is rejected at a significant value of 5%. Corrections could be performed to overcome the violation of sphericity.

Regarding the violation of the normally distributed data assumption (p-values <0.05), the analysis of variance between time points was again conducted, although with the Friedman test (a non-parametric test) [24]. The null-hypothesis and the alternative hypothesis, for this test, are the same as the ones formulated for the repeated measurements ANOVA test. The Friedmann's test is described by Equation 2 [24].

$$\chi 2 = \frac{12n}{p(p+1)} \sum_{j=1}^{p} \left\{ \overline{r_j} - \frac{1}{2}(p+1) \right\}^2 \qquad (2)$$

where $\overline{T_j}$ is the mean rank of the jth time point, p is the number of ranks (time points) and n the number of rows (total number of samples). If $\chi 2$ is too high, than the mean ranks differ significantly [24].

The statistics for this test, $\chi^2(4) = 3.800$, p-value = 0.434 (> 0.05), does not let to reject the null hypothesis of the COF mean values being equal through all the time points. This outcome enables to consider the mean values of COF, through all the time points, for further analysis.

3.4   Effects of factors on the response variable (COF)

Problem. The aim of this statistical analysis was to understand how the diamond particles size and the type of technology used in each sample affect the COF. Having this in mind, both levels of each factor were considered and the mean COF over time from the three trials (**Table 5**) was tested for each combination of factors.

Table 5. Means of COF for each of the three trials over time, for the four samples.

		Diamond particle size (factor A)	
		0,1-0,5 μm	40-60 μm
Type of Technology (factor B)	HP	0.46897; 0.43269; 0.44838	0.11947; 0.13091; 0.13196
	LS	0.58369; 0.54460; 0.49262	0.13831; 0.15715; 0.11585

Resolution. This two-factor experiment has 12 observations (2 diamond particles sizes x 2 technologies of sintering x 3 trials). In order to test the significance of the effect of each factor, the first attempt was to apply the Analysis of Variance (ANOVA) method with a two-factor factorial design. Firstly, for the application of this method, its assumptions must be satisfied, namely the response variable (mean of COF over time) has to be normally distributed; homoscedasticity has to be verified; as well as randomness of the data [23]. To check the normality of the data, the Kolmogorov-Smirnov (K-S) test was used.

Accordingly, the K-S test points to the same conclusion, as $D(12) = 0.290$, p-value = 0.006 (p-value < 0.05), therefore rejecting the null hypothesis.
Given that one of the ANOVA assumptions is not fulfilled, it was necessary to resort to another statistical method, in this case, a nonparametric method. Since the response variable presents different variabilities for each combination of factors' levels, a test to compare the distribution of two independent samples was chosen - the two-sample K-S test. The respective statistic, $D_{m,n}$, is calculated as presented in equation 3 [25].

$$D_{m,n} = \sup_{|x|<\infty} \left| \hat{F}_{1m}(x) - \hat{F}_{2n}(x) \right| \tag{3}$$

where *m* and *n* are the samples sizes, x is the response variable; and $\hat{F}_{1m}(x)$ and $\hat{F}_{2n}(x)$ are the empirical distribution functions obtained from each sample.

Regarding diamond particles size, by applying this method, it was possible to conclude that the distribution of COF across the 0.1-0.5 μm and 40-60 μm diamond particles is not the same, since the software returned the results of D(12) = 1.732, p-value (2-sided test) = 0.005. In contrast, the results that concern to the type of technology point to a similar distribution of COF across HP and LS technologies, as D(12) = 0.866, p-value (2-sided test) = 0.441.

Therefore, on the basis of the two-samples K-S test, it is possible to conclude that the diamond particles sizes used in the sintering process produced a statistically significant impact in COF (p-value < 0.05), since the two particles sizes induced a statistically different effect on the response variable for both technologies of sintering. Contrarily, the type of technology did not affect COF in a statistically significant way (p-value > 0.05).

3.5 Effects of factors on the response variable (SWR)

Problem. The main goal of this statistical analysis was to study the effects of the two diamond particle sizes (0.1-0.5 and 40-60 µm) and two technology types (Laser Sintering and Hot Pressing) on the wear of the sample. Table 6 presents the two levels of each factor considering the combination of factors.

The negative value of wear observed for the HP samples with particle sizes 40-60 µm means that the counter body had transferred mass to sample. The null value of wear observed for the same particle size using laser sintering technology means that there is an equilibrium between the mass transferred from counter body to sample and from to sample to counter body.

Table 6. SWR values for each of the three trials, for the four samples.

| | | Diamond particle size (factor A) | |
		0,1-0,5 μm	40-60 μm
Type of Technology	HP	3.71e-05; 2.41e-05; 2.08e-05	4.42e-06;0.00e+00; 2.65e-06
	LS	1.20e-05; 1.28e-05; 1.06e-05	-1.09e-06; 01.75e-0;61.09e-06

Resolution. The Analysis of Variance (ANOVA) method was performed in order to test the significance of the effect of particle size and technology type. However, some assumptions [response variable (wear) normally distributed, homoscedasticity and randomness of data] must be verified. The normality of the data was checked by performing the Kolmogorov-Smirnov (K-S) test.

Considering a level of significance (α) of 5%, the null hypothesis is not rejected [D(12) = 0.198 and p-value = 0.200 (p-value > α)] and therefore the wear data follows a normal distribution. The constant variance (homoscedasticity) was checked by performing the Levene's test. The results of Levene's test allowed to confirm the

homogeneity of variance, once the obtained p-values were of 0.15 and 0.20 for different technologies [D(12) = 8.590] and diamond particle sizes [D(12)= 7.573], respectively.

Considering a full factorial model, the analysis of interactions and factors was performed.
The effect of a factor (particle size or technology type) indicates a variation in the response variable (wear) by a change in the levels of particle size or technology type. An interaction between the two factors is verified when the effect on one factor depends on the condition of the other factors.

The interaction between particle size and type of technology (H_{03}) was observed since for $F(1,8) = 6.872$ the p-value obtained was 0.031 (< 0.05), so the null hypothesis is rejected. Therefore, it can be concluded that the interaction between particle size and technology type affects the SWR. Since an interaction was observed, the analysis of main effects does not explain correctly the effect of factors on response variable.

The high value of R squared ($R^2 = 0.891$) obtained on the ANOVA test means that the relation between the response variable and levels of factors and the interaction are well explained by the model. The same conclusion can be taken by observing the corrected value (p-value ~ 0.00 < 0.05) on ANOVA table once there is a significative statistical relation. Additionally, the low value of the error (2.067e-11) spelch the variability of the residuals, which correspond to random errors that models cannot explain. Therefore, this allows to conclude that, in fact, there is an interaction.

Fig. 5 presents the interaction plot that displays the fitted values of the wear variable (dependent variable) on the y-axis and the particle size values (0.1-0.5 µm and 40-60 µm) on the x-axis. The two lines (red and blue) represent the technology types (Hot Pressing and Laser Sintering). The different slopes propose that there is an interaction effect and the p-value for the particle size / technology type confirms the mentioned previously.
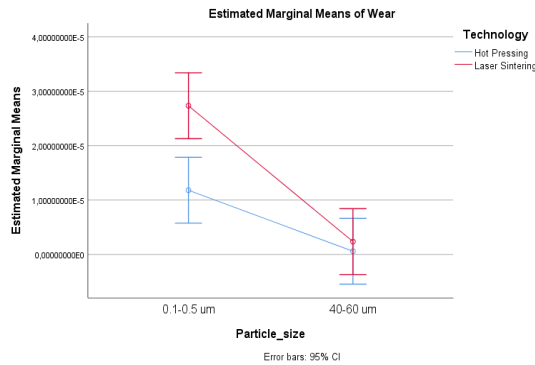
Fig. 5. Interaction effect between particle sizes for two technologies considered

From the distances between the segment edges, it is possible to see that, for smaller particles, there are significative differences between technologies, while for bigger particles this is not verified, suggesting that the behavior of the factor levels changes with type of technology used.

Additionally, the normality of the residuals was checked by performing the K-S test.

Considering a level of significance of 5%, the null hypothesis is not rejected [D(12) = 0.215 and p-value = 0.102 (p-value > 0.05)] and therefore the wear data follows a normal distribution.

The homoscedasticity of the residuals was checked by performing the Levene's test. The plot of residuals against estimated values of wear is shown in Fig. 6. The results of Levene's test confirm the homogeneity of variance. A p-value of 0.102 for D(12) = 0.215 was obtained and therefore, the null hypothesis is not rejected.

The homogeneity of variance is confirmed by a satisfactory pattern. However, the Fig. 6 reveals a funnel pattern for residuals, which means that there are anomalies.
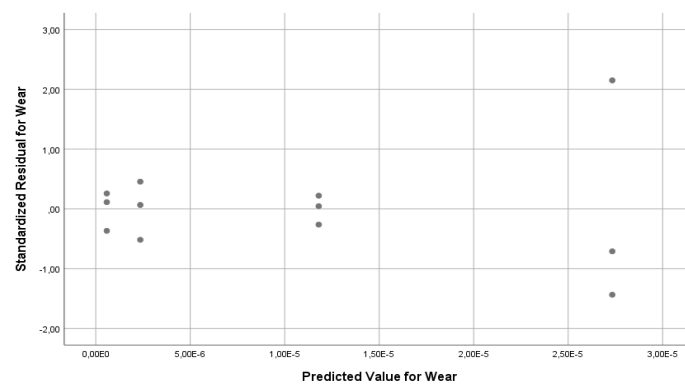
Fig. 6. Plot of residuals against estimated values of SWR.

In order to understand the anomalies verified in the previous graph, a boxplot of two diamond particle sizes and two type of technologies (Fig. 7) was executed. The graph allowed to conclude that the non-satisfactory pattern might be due to the large dispersion observed for particle size of 0.1-0.5 µm and LS technology, when compared to the other conditions. It is also possible to conclude about the absence of outliers.
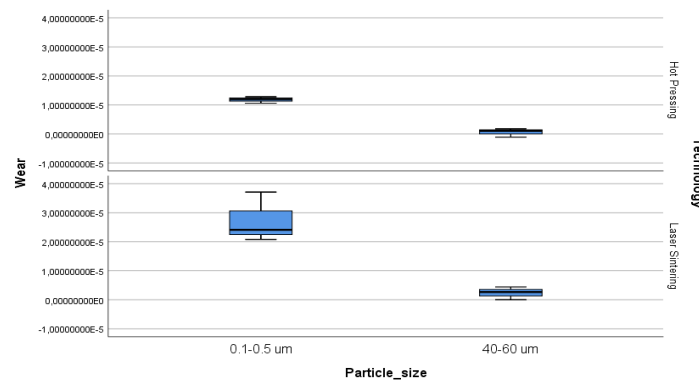
Fig. 7. Boxplot of two diamond particle sizes and two type of technologies

As mentioned during ANOVA table analysis, the high value of R^2 (0.891) revealed a well explained model. The corrected model with p-value lower than significance level proves a significative statistical relation and the Levene's a variance homogeneity. So, considering these points and the variability on boxplots, the anomalies found on residuals should not be considered relevant.

4 Conclusions

The modification of the tribological properties of laser textured 316L stainless steel reinforced with a CuCoBe-diamond composites was investigated in this work. By performing an initial visual analysis of the data, it was possible to predict some of the statistical inferences described. However, a robust statistical study and an appropriate experimental planning require the utilization of a statistical software as the IBM® SPSS.

In the first part of this analysis, in order to access the possible variability of COF values through time, repeated measures ANOVA was investigated. The violation of sphericity and normally distributed data assumptions lead to the use of the Friedmann's test. The results of this non-parametric test suggested not to reject the null hypothesis of equal mean values for COF through time ($\alpha = 5\%$). Therefore, subsequent analysis with the mean values of COF through all time range are suggested to be performed in future investigations on this theme. In addition, according to the two-samples K-S test, the different diamond particle sizes produced a statistically significant impact on the COF, whereas the type of technology did not affect this parameter in a statistically significant way.

Finally, regarding to the effects of the factors on the response variable SWR, the assumptions (normality and constant variance) were verified at both data and residuals analysis. The high value found for R squared ($R^2 = 0.891$) obtained on the ANOVA test indicated that the relation between the response variable and levels of factors, as well as the interaction were well explained by the model. In addition, the interaction between particle size and type of technology was verified, so the analysis of main effects did not explain correctly the effect of factors on the response variable.

Acknowledgements

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