Engineering and Applied Sciences
2025, Vol. 10, No. 3, pp. 38-45
https://doi.org/10.11648/j.eas.20251003.12

Research Article

Microabrasive Wear Test Procedure for Characterization of High Hardness Ferrous Materials

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Abstract

The ball cratering microabrasive wear test is a method whose feasibility in wear resistance characterization of metallic materials has been demonstrated in numerous investigations, however, there is still no standard that establishes the procedure for its application and the test requirements. This leads to the need to establish, through prior experimentation, the test procedure suitable for specific applications. In the present work, a microabrasive test procedure was obtained, aimed at the characterization of high alloy and hard ferrous materials, based on the quality of the wear mark, the behavior of the crater diameter with respect to the test time and the results dispersion. It was demonstrated that, by using alumina as the abrasive material, with a concentration of 10 g per 100 ml of water, a dropping frequency of one drop every 5 seconds, a test force of 0.27 N, a shaft rotation speed of 80 rpm and a test time of 10 minutes, reliable measurements are obtained in a permanent wear regime, which shows the importance of adapting this test parameter to the type of under-study material. It was also found that there is a correspondence between the hardness and the microstructure of the material with the average size of the wear crater and the dispersion of results.

Keywords

Abrasive Wear, Ball Cratering Microabrasive Wear Test, Wear Resistance

1. Introduction

Abrasive wear is defined by American Society for Testing and Materials, in ASTM G40 standard, as the type of surface damage due to hard particles or hard protuberances forced against and moving along a solid surface [1].

León, et al agree that the nature and extent of abrasive wear is dependent on a number of factors, including material microstructure, the kind of abrasive particles and its characteristics (size, shape, hardness), the magnitude of stresses in the system components, the contact frequency of the abrasive particle with the surface, relative movement, temperature and the effect of chemicals [2]. These factors interact to produce a complex tribological system, altering the intensity of the process and thus the rate (Q), coefficient (k) and wear volume (V). Hence, it can be argued that wear resistance is not an intrinsic property of the materials, but depends on the set of conditions that define the system, and therefore on the method and procedure used for its study.

In recent decades, a new configuration of equipment called "ball cratering microabrasive wear machine" has been used to characterize the materials behavior under abrasive wear conditions, initially applied to the coating thicknesses measurement, but with recognized results in the characterization of

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Received: 9 January 2025; **Accepted:** 30 April 2025; **Published:** 6 June 2025

ferrous materials [3-8].

In this case, due to the contact between a rotating sphere and the test sample surface, under the action of an abrasive paste at the interface, a hemispherical crater is generated as a wear mark, whose dimensions allow predicting or estimating the relative behavior of the material. There are different configurations of this type of test (Gee, et al.; 2003), the one that stands out for its simplicity is which applies the "free ball" principle (Figure 1) [9]. In this case, the rotation of the sphere occurs due to the rotation of an axis, which drives it by the action of friction with the edges of a notch.

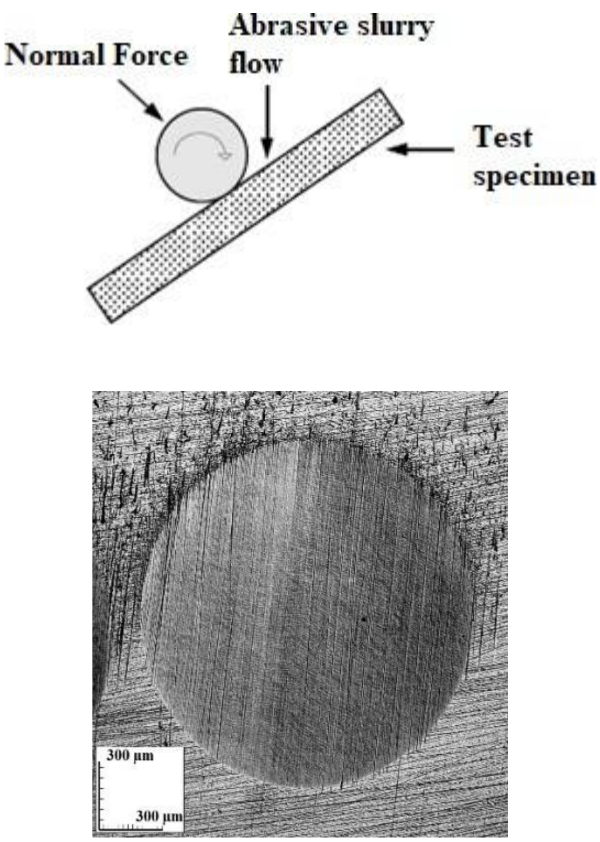


Figure 1. *"Free ball" microabrasion tester. a) Principle of operation, b) typical wear crater image [3].*

Cozza, et al have demonstrated that by means of the ball cratering microabrasive wear test, it is possible to obtain "two-body", "three-body" and a combination of both abrasive wear modes, for different load/concentration conditions of the abrasive paste, besides evidencing high stress abrasive wear in some applications [4, 10]. On the other hand, the small size of the abrasive particles causes the wear craters to present defined edges, thus achieving high precision of measurements [3].

This test is standardized for the study of ceramic coatings under the British Standard (BS) as BS EN 1071-6 and International Organization for Standardization (ISO) as ISO 26424 standards, but there is no standard for its application as a method for characterizing the wear resistance of metallic materials [11, 12]. This causes that in the different studies where the application of this method is reported, there is no systematicity of criteria that support the selection of the test variables, which evidences the need to support the selection of the test regime parameters by previous research on the materials under study.

Adachi & Hutchings obtained a map of the abrasive wear modes in which this is manifested in dependence of the load and the concentration of the abrasive material, for which they used silicon carbide particles with grain size in the order of 4 µm [13]. This result is applicable to the materials studied, as long as the characteristics of the abrasive paste and the test parameters are maintained within the ranges used in these experiments. The same is true for other investigations, where a great diversity of materials and a wide range of parameters are used, as evidenced in a review reported by Rodríguez [14]. In addition to silicon carbide, other materials are used as abrasives, such as quartz, alumina, diamond powder and zirconium oxide, in different grain size and concentration.

On the other hand, to reliably characterize the material behavior in a given tribological system, the wear process must occur under permanent regime, which is reached when the surface settles and the irregularities effect disappears. This ensures that the intensity, rate and coefficient of wear remain constant [15]. Hence, the moment at which steady-state wear begins is determined by the surface finish of the sample, the abrasive material, its grain size and concentration, as well as the test parameters, which implies the need to establish operating techniques depending on the material to be studied, the set of test parameters and the characteristics of the abrasive material.

The present study aims to obtain a "free ball" microabrasive wear test procedure for the characterization of high alloy and hard ferrous materials, used in applications where abrasive wear conditions predominate, based on the determination of test parameters that guarantee the quality of the measurements, in a permanent wear regime.

2. Experimental Procedure

2.1. Selection of Test Materials

Méndez, et al state that among the materials most commonly used to face working conditions where abrasive wear predominates are carbide-based alloys, mainly chromium-alloyed white cast irons [16]. Other materials that provide high resistance to abrasion are steels with martensitic microstructure, high manganese steels (Hadfield) after impact hardening, as well as several types of Ni, Cu or Co base alloys with carbide-forming elements in their composition. Based on the above, the materials selected for the study are white cast iron ASTM A 532 class I type A and a tool steel, heat treated, classified by the Russian Government Standard (GOST), especificaly GOST 5950 standard, as 9XBG, [17, 18]. A sample of low carbon steel, classified according to the

American Iron and Steel Institute (AISI) as AISI 1015 steel. was also included in the study in order to contrast the results in terms of the correlation of the material characteristics with its behavior against microabrasive wear, and with them to analyze the sensitivity of the characterization method [3]. The chemical composition of these materials, established in the classification standards, is shown in Table 1.

Table 1. *Chemical composition of steels used for testing [17-19].*

Stand./ Classif.	Chemical composition (% by weight)							
	C	Mn	Si	Cr	Mo	V	Ni	Others
ASTM A 532	3.0-3.6	1.3	0.8	1.4-4	1.0	-	3.3-5.0	-
GOST 9XBG	0.85-0.95	0.9-1.2	0.1-0.4	0.5-0.8	≤0.2	≤0.15	≤0.4	W: 0.5-0.8 Ti ≤0.03 Cu ≤0.3
AISI 1015	0.13 - 0.18	0.3 - 0.6	0.35 máx.	-	-	-	-	-

Note: P and S contents do not exceed 0,040% and 0,050% for all materials.

These samples were characterized by metallographic technique, for which it was necessary to prepare the surface by grinding and polishing, according to the procedure established in ASTM E3 [19]. The chemical attack was performed by immersion, using Nital (1%) for the AISI 1015 steel and GOST 9XBG steel samples, and Vilella for the white cast iron, according to ASTM E407 [21].

The microstructure was observed using a Neophot 32 metallographic optical microscope. In addition a Heeckert hardness tester was used for hardness measurements, with a load of 1 kgf, for 10 seconds, based on ASTM E384 [21].

2.2. Selection of Ball Cratering Microabrasive Wear Test Parameters

The ball cratering microabrasive wear test with “free ball” equipment used, as well as the operating technique, were both developed and validated by López, et al [22]. The abrasive material selected was calcined alumina, of "AnalaR®" brand and English origin, with a particle size less than or equal to 1 µm. The use of this abrasive for the study of high hardness and abrasion resistance materials is based on its high hardness (2100 HV0.2), reported by Badisch & Mitterer [23]. In addition, Stachowiak, et al. [8] state that alumina particles have high angularity, higher than silica sand and quartz, which favors the wear test severity. This is used in a concentration of 10 g of alumina per 100 ml of distilled water, used with excellent results in works by López [22].

For setting of the other test variables, previous experiences in applications of “free ball” microabrasive test on metallic materials, reported in the literature and summarized in the work of Rodríguez, were used as starting point [14]. Based on this, an abrasive paste dripping frequency of 1 drop every 5 s was used, which allows accelerating the wear process, considering the high hardness of the materials to be studied and the importance of reducing the experimentation time to increase the efficiency of the test procedure.

As for the normal force, the value of this parameter for the free rotating sphere test is determined by the weight of the ball and the maximum angle of inclination with respect to the vertical plane. Generally, this parameter does not exceed 1.1 N for tests with free rotating sphere. On the other hand, Cozza [4] states that high sample inclination angles should be used to avoid ball slippage in its interference with the axis. Considering the above, an angle of 70 was selected. The equipment to be used employs 25.4 mm diameter balls, made of AISI 52100 steel, with a mass of 0.067 kg, which exerts a normal force on the test surface of 0.27 N.

The shaft rotation speed is one of the parameters generally reported in the works where this wear test is used. In the different works analyzed by Rodríguez [14], it can be seen that the shaft rotation speed varies in a range between 37.5 and 150 rpm, for the equipment with free rotating sphere, being in most cases below 80 rpm. Cozza, et al. justify the selection of low rotation speeds (37.5 rpm) because this eliminates the hydrodynamic effect [24]. On the other hand, Bethke states that for free ball equipment, the tendency of the ball to slide on the shaft increases as the speed increases [25]. Both factors can lead to an apparent decrease in wear as the ball speed increases, which affects the reliability of the test results. Given the above, a shaft rotation speed of 80 rpm was used.

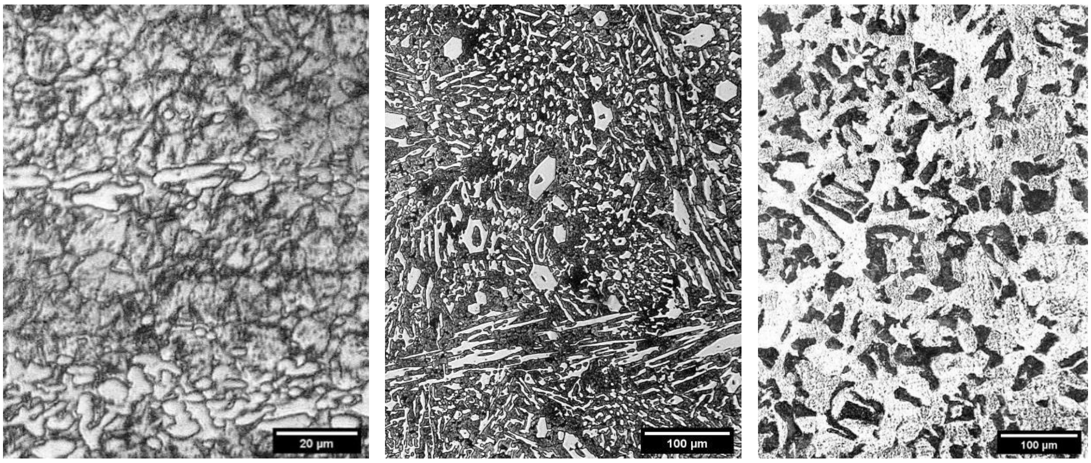
As for the sliding distance and test time, these parameters are directly linked, and the values of these parameters are increased for materials with high resistance to abrasive wear, in order to test in a permanent wear regime and obtaining craters with sharp contours, which allow to be measured with quality. Based on the above, the test times selected for the study were 5, 10 and 15 minutes, corresponding to distances of 63.8 m, 127.6 m and 191.4 m, respectively.

Measurement of the wear crater diameters was performed using a portable microscope (read- out), low magnification (24x). The experiments were repeated three times. Statgraphic Centurion V18 software was used for the statistical evaluation of these results, as well as to obtain the wear behavior models as a function of time. The crater diameter was used as a representative magnitude of the wear resistance, considering that it allows a more precise evaluation of the property than the wear volume, as a result of a smaller propagation of the error [3].

3. Results and Discussion

3.1. Results of the Metallographic Characterization of the Under-Study Materials

Figure 2 shows the microstructures of the materials selected for the study. In the case of the GOST 9XBG classification steel, no acicular shaped phases typical of the quenching heat treatment are observed, with the magnification used to visualize the microstructure (Figure 2a). However, the hardness study yielded an average value of 554.7 HV, which allows to affirm the material has been hardened by heat treatment. This gives it favorable properties against abrasive wear, agreed the high hardness of the phases of which it is composed.



a) b) c)

Figure 2. *Microstructure of the under- study materials, a) GOST 9XBG steel, b) white cast iron ASTM A 532 c) AISI 1015 steel.*

Figure 2b shows the microstructure of white cast iron with hypereutectic morphology, which is composed of primary carbides of M7C3 type (hexagonal cross-section bars), surrounded by a zone of eutectic appearance composed of secondary carbides and metallic matrix, typical of this kind of material [3, 26, 27]. The wear resistance of hypereutectic white cast irons is determined by the effect of primary and eutectic carbides, given by their size, shape and chemical composition, as well as the microstructure of the matrix. The average value obtained in the hardness test to this sample is 555.7 HV, similar to that reported for this type of material [28].

As expected, the microstructure of the steel with AISI 1015 classification, shown in Figure 2c, is composed by ferrite and perlite, similar to that reported for this type of material [3]. The hardness obtained, 110.7 HV, is within the range reported for this steel in the annealed condition, which is corroborated given the equiaxial morphology of the grains in the microstructure [29].

3.2. Analysis of Wear Test Results

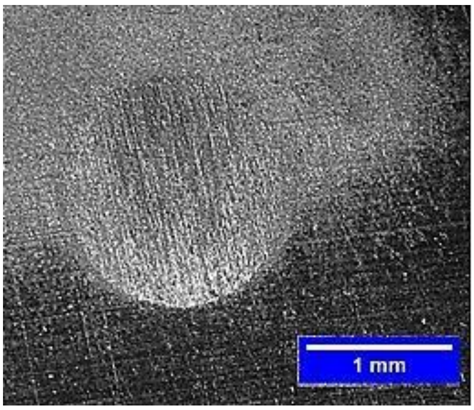


Figure 3. *Worn surface appearance of ASTM A 532 white cast iron sample, after 15 minutes of testing.*

The typical appearance of the wear craters obtained in the white cast iron ASTM A 532, tested with a time of 15 minutes, is shown in Figure 3, where the wear marks can be seen in form of parallel grooves, showing that the abrasive wear mechanism occurred by the two-body mode. Similar appearance was manifested in the other experiments, for all three materials. In all cases, adequate contour sharpness was obtained, which is evidence that the permanent wear regime was reached during the test [30].

The wear results obtained in the microabrasive test, for the test times of 5, 10 and 15 minutes, are shown in Table 2. The magnitude of the mean diameter of the crater (dm) was used as an evaluation parameter, since this allows a more accurate assessment of the abrasive wear resistance property of the material than the wear volume, as a result of smaller error propagation [3].

Table 2. *Wear crater diameter in the under-study materials and the variation coefficient of measurements.*

Parameters	**Test time, t (min)**		
	5 min	**10 min**	**15 min**
GOST 9XBG Steel			
Average diameter (dm) (mm)	1.13	1.27	1.38
Variation Coefficient (%)	2.22	1.14	1.82
ASTM A 532 White Iron			
Average diameter (dm) (mm)	1.07	1.25	1.34
Variation Coefficient (%)	3.58	2.00	1.08
AISI 1015 Steel			
Average diameter (dm) (mm)	1.375	1.44	1.59
Variation Coefficient (%)	1.82	1.00	0.91

To ensure that the results are significantly different, a sample comparison analysis by means of the Analysis of Variance method (ANOVA), was performed with the crater diameters taken at the three test times for each studied material, which showed in all cases that there is a statistically significant difference between the means of the crater diameters for the three test times (5, 10 and 15 min.).

Another aspect to highlight is the behavior of the coefficient of variation of the crater diameter in the different test times for the materials tested. Taking as a reference what is established in the ASTM G65 standard, values lower than 7 for this statistic is a reflection of the repeatability of the wear test and therefore of the quality of the measurements.

As can be seen (Table 2), this condition was satisfied in all cases. However, it should be noted that as the hardness and wear resistance of the material increases, the dispersion of the results increases too, especially in the case of the lowest test time (5 min.), which indicates that the test should be carried out with times longer than this, in order to optimize the reliability of the method.

Cozza, et al state that when the wear presents a linear variation with the test time (or sliding distance), the permanent wear regime has already been reached [31]. In order to verify whether this occurred in the tests, the curves of the crater diameter behavior as a function of time (t) were obtained for the three materials tested (Figure 4).

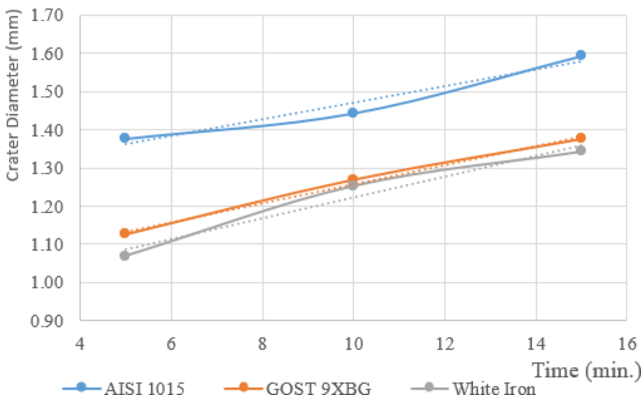


Figure 4. *Wear behavior of the materials tested.*

Figure 4 indicates the superior wear resistance behaviour of the ASTM A 532 white cast iron, despite having a similar hardness to the heat-treated GOST 9XBG steel. This corresponds with Wang, et al. and Coronado, et al., who consider that hardness does not describe abrasive wear resistance in materials of different alloy systems [32, 33]. This behavior of wear resistance in the hypereutectic white cast iron sample is justified by Chatterjee & Pal, who explain it due to the presence within the microstructure of high hardness alloyed carbides (M_7C_3: 1600- 1800 HV), which act as effective barriers to the effect of abrasive particles [34]. In the case of GOST 9XBG tool steel, its favorable behavior is due to the high hardness resulting from the heat treatment of this material, as proved in the hardness test.

The graph in figure 4 shows that the highest wear occurred in AISI 1015 steel. This is an expected result given the unfavourable behaviour of carbon steels under abrasive wear conditions, which is due to the low hardness of its constituent phases, several times lower than abrasive particles.

The equations for the mean crater diameter (dm) as a function of test time (t) are given below (equations 1, 2 and 3) for the three materials.

$$d(GOST\ 9XBG) = 1.008 + 0.025 \cdot t \quad (1)$$

$$d(White\ Iron) = 0.949 + 0.027 \cdot t \quad (2)$$

$$d(AISI\ 1015) = 1.254 + 0.022 \cdot t \quad (3)$$

In all cases the quality of the linear models was supported by the results of the ANOVA test, demonstrating a statistically significant relationship between crater diameter and test time with a confidence level of 95.0%, model fits above 90.0% and correlation coefficients above 0.96, indicating a relatively strong relationship between the variables. In all cases the standard error of the estimate does not exceed 3% and the mean absolute error is less than 2%, indicating a minimum dispersion of the sample. From the above, the linearity of the behaviour of the wear crater diameter with respect to time is evident in the three materials studied, in the three test times used (5, 10 and 15 min.), which indicates that the wear phenomenon occurs in a stationary or permanent regime, the period in which the measurements should be made for the characterisation of this materials, and therefore endorses the appropriate selection of the test times. However, considering the relative dispersion of the data set, given by the coefficient of variation (table 2), it can be seen that this becomes less than 2% for times longer than 10 minutes, which reflects the quality of the measurements of the study method. Considering also as a criterion the efficiency in the experimentation, it can be considered that the use of 10 minutes of test time, together with the parameters of the selected regime and the employed operative technique, is adequate for the study of high alloy and hardness ferrous materials.

Finally, another aspect standing out when analysing equations 1, 2 and 3 is the similarity of the slopes of the curves up to the second decimal place (0.02), which is expected since the wear rate depends on the test parameters and not on the test material. This result supports the reliability of the wear measurements obtained by the "free ball" microabrasive wear test technique applied to ferrous materials of high hardness and resistance to abrasive wear and the appropriate selection of the test parameters used for this purpose.

4. Conclusions

1) The ball cratering microabrasive wear test using "free ball" principle, on high alloy and hard materials, using alumina (concentration of 10 g/100 ml water) as abrasive material, with a frequency of one drop every 5 seconds, a test force of 0.27 N, a shaft rotation speed of 80 rpm and a test time of 10 minutes, allows reliable measurements to be obtained in a permanent wear regime.
2) In the conducted tests, as the hardness and wear resistance of the material increases, the dispersion of the results rises. From the test time of 10 minutes onwards, the coefficient of variation of the measurements did not exceed 2% for any of the tested materials, which shows the importance of adapting this test parameter to the type of under-study material.
3) The results of worn material volume loss on the samples of ASTM A 532 class I type A white iron, GOST 9XBG tool steel and AISI 1015 carbon steel, correspond proportionally with the relative hardness of these materials and the composition of their microstructure, thus demonstrating the superiority of the alloyed white irons against the abrasive wear mechanism.

Abbreviations

ASTM	American Society for Testing and Materials
GOST	Russian Government Standard (государственный стандарт)
BS	British Standard
ISO	International Organization for Standardization
AISI	American Iron and Steel Institute
ANOVA	Analysis of Variance Method

Author Contributions

Tamara María Ortiz Méndez: Conceptualization, Methodology, Project administration, Supervision, Writing – original draft, Writing – review & editing

Dayán Montero Madán: Data curation, Formal Analysis, Investigation, Software, Validation, Visualization

Amado Cruz Crespo: Conceptualization, Methodology, Supervision, Writing – review & editing

Jorge Víctor Miguel Oria: Data curation, Formal Analysis, Investigation, Visualization

Conflicts of Interest

The authors declare no conflicts of interest.

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