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Oxidation and abrasive wear of Fe–Si and Fe–Al intermetallic alloys

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Abstract In the present work, intermetallic alloys Fe–Si and Fe–Al (Fe_3Si–C–Cr and Fe_3Al-C), produced by induction melting, were evaluated regarding their oxidation and abrasive resistance. The tests performed were quasi-isothermal oxidation, cyclic oxidation, and dry sand/rubber wheel abrasion. As reference, the ASTM A297-HH grade stainless steel was tested in the same conditions. In the oxidation tests, the Fe–Al based alloy presented the lowest oxidation rate, and the Fe–Si based alloy achieved the best results in the abrasion test, showing better performance than the HH type stainless steel.

Introduction

Intermetallic-iron based alloys have been quite studied due the oxidation and good mechanical properties at elevated temperatures. Furthermore, they present low cost and low density comparing with other iron-based alloys and thus are candidates to replace stainless steels in many applications [1]. Their corrosion resistance is due to the oxide adherent layer formation on the surface, Al_2O_3 for Fe_3Al and SiO_2 (hydrated) for Fe_3Si, occurring because of the high content of Al and Si in the alloys, around 15 wt% [2, 3].

In the last three decades, several oxidation studies have been performed on FeAl and Fe_3Al alloys [2, 4, 5]. Regarding Fe–Si, mainly because the material's brittleness, few studies are found. The main and more recent references are from Kim et al. [6], who performed mechanical and corrosion tests, and from Castro et al. [3, 7].

Regarding wear properties, the aluminide intermetallics have been mainly studied in the 1990s and after [8–10], but, for Fe–Si based intermetallics, few information can be found in the literature. The interest on the Fe–Al alloys is mainly because of its high temperature properties, such as oxidation resistance, yield strength increase with increasing temperature up to 650 °C, high work-hardening rates, and no significant content of high cost elements. In more severe erosive wear and erosion applications they could replace the most used alloys, as Co-based, high manganese stainless steels and other chromium-containing alloys [8]. A study on Fe–Si, evaluating the Cr influence in the slurry abrasive wear resistance, has shown an improvement, but no comparison with other materials classes has been made [7].

The main disadvantages found for Fe–Al and Fe–Si alloys are the low-creep resistance and the brittleness, because of the low thoughness. For Fe_3Si this brittleness is intrinsic of the material and, for Fe_3Al, hydrogen embrittlement can also occur [1, 6].

The aims of the present work were to evaluate both ferrous intermetallic alloys, with aluminum and with silicon, under quasi-isothermal oxidation, cyclic oxidation, and dry sand/rubber abrasion test. Furthermore, the performance of the alloys was compared with the results found for HH austenitic stainless steel.

Materials and methods

In the present study, quasi-isothermal oxidation, cyclic oxidation and dry sand/rubber wheel abrasion tests were applied on the materials: Fe–14.5%Al–1.1%C,

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Fe–14.5%Si–0.75%C–4%Cr and ASTM A297 HH austenitic stainless steel for comparison. The intermetallic alloys choice was made based on the literature, aiming a simple and cheap chemical composition. For the Fe–Si alloy, the chromium presence was considered because of earlier studies [3, 7], where Cr addition improved the wear and corrosion resistance.

The quasi-isothermal oxidation test, also known as discontinuous oxidation, was performed at 800 °C. This kind of test differs from isothermal oxidation tests because the samples are cooled before weighing. A rate of 30 °C/min was used to heat the samples, and the cooling occurred in calm air during short periods just to evaluate the material mass change. After each weighing, the samples were immediately returned into the furnace and the periods between each cooling were chosen arbitrarily, without an exact frequency, which in turn distinguish such experiments from cyclic oxidation tests [11–13]. For cyclic oxidation tests, a special furnace has been used which allows automatic and controlled specimens exposition to calm air. The specimens were heated up to 800 °C with the same heating rate as above. Each cycle was performed by holding the sample 1 h inside the furnace followed by 10 min of exposure to calm air. These controlled periods of heating and cooling are the main difference between the cyclic and quasi-isothermal tests. Furthermore, in the cyclic test the weighings do not occur for each cooling, they are fast performed during few cooling cycles of the entire test. Therefore, the heating/cooling frequency is virtually constant.

The dry sand/rubber wheel abrasion tests were in accordance with the ASTM G65 standard recommendations. The evaluated specimens were under two conditions: as-cast and as-oxidized. The abrasive flow during the tests was 400 g/min, the load applied was 130 N and 2,000 wheel revolutions were used.

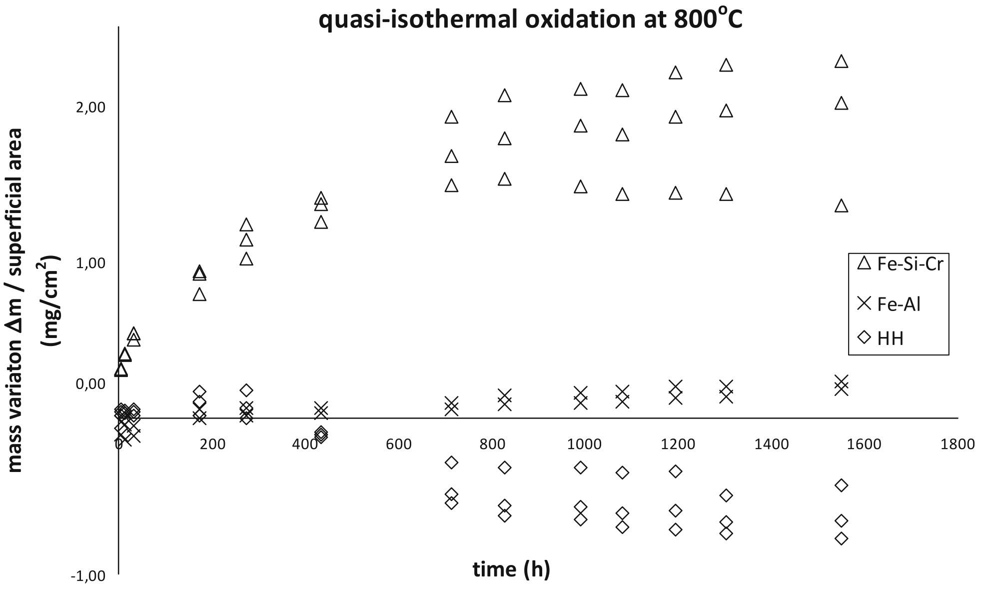
Results and discussions

Oxidation tests

Figure 1 shows the quasi-isothermal test results, presenting the mass variation by the superficial specimen area against the exposure time. The quasi-isothermal term is used because the short cooling times used for mass measurement during the tests. In fact the samples should remain inside the furnace during weighing in order to produce a true isothermal experiment. In the quasi-isothermal tests, the Fe_3Al alloy presented the better results, with less mass variation. On the other hand, the Fe_3Si alloy presented the higher mass variation (mass gain), and an inverse behavior was observed for HH steel, which had mass loss.

These behaviors are explained by the surface oxide layer formation. For Fe_3Al, the surface oxide quantity almost does not change, because the alumina (Al_2O_3) is stable and adherent forming a compact and thin layer with low growth rate. For Fe–Si, the mass variation occurs due to the SiO_2 oxide layer increasing, and after almost 800 h, the mass gain rate becomes quite low, with a parabolic profile, showing that the material does not suffer catastrophic oxidation. For HH steel, the oxide layer formation and spallation occur with a fairly slow rate. This behavior is due to the cooling step for mass measurement. During this process, the different coefficient of thermal expansion, between the oxide and the base material, generates the

Fig. 1 Mass variation on the quasi-isothermal oxidation test at 800 °C, comparing Fe–Si, Fe–Al, and HH alloys



layer oxide spallation. This fact is undesirable in applications where the component dimensions should remain nearly constant while suffering heating and cooling cycles, what is quite usual in many sorts of industrial equipments.

Figure 2 shows the cyclic oxidation results of the three materials. The behaviors presented were similar comparing with quasi-isothermal oxidation and the mass variation was also close to that results. Nicholls and Bennett [11], in a review, cited three steps in cyclic oxidation for materials with alumina and chromia layers as protection agents, called “birth”, “life” and “death”. Summarizing, the oxide layer grows in parabolic or sub-parabolic oxidation kinetics with mass gain (“birth”), the layer spalls and new oxide forms healing the regions and creating depleted zones of forming protective oxide elements, with mass loss (“life”), and when these depleted regions reach a critical level, the base material suffers fast and catastrophic oxidation, a phenomenon known as “breakaway oxidation” (“death”). Comparing this information with the obtained results we can verify that no intermetallic started to spall. The HH steel had loss of mass, in a slow rate, suggesting the material was far from the “breakaway oxidation” stage but towards that direction. One of the important factors in this case is the 800 °C temperature, which can be considered low for these materials, making the cyclic oxidation a slow process.

Concerning to the different kind of oxidation tests, some studies with stainless steel [14] and intermetallic materials [12, 13], compared isothermal, quasi-isothermal, and cyclic oxidation tests. Toscan et al. [14] found, for ferritic stainless steel, the same behavior under isothermal and cyclic oxidation tests, always observing gaining of mass. For austenitic stainless steel they observed loss of mass under cyclic oxidation, probably due to oxide layer spallation during the cooling step as observed for the HH steel, which lost mass under the same condition in the present study. However, they also verified mass gain for austenitic stainless steel under isothermal oxidation, in contrast with the present quasi-isothermal tests. Vojtěch et al. [13] evaluated two Ti–Al intermetallic alloys and obtained mass gain under the isothermal condition. On the other hand, under quasi-isothermal tests, one alloy presented mass gain and other mass loss. The alloy that lost mass gained more mass at the isothermal condition, suggesting that alloys with higher mass gain have more mass loss tendency under quasi-isothermal conditions, due to the thicker oxide layer, although this fact depends on the oxide adhesion on the base material. In the present study, considerable mass gain was found for the Fe–Si intermetallic but no spallation tendency was observed, at least for the number of cycles studied.

Braun et al. [12] evaluated intermetallic Ti–Al. Initially, they observed mass gain, under quasi-isothermal and cyclic oxidation, and, after a period of time, in both cases oxide layer spallation occurred with mass loss. For the quasi-isothermal experiment the spallation happened after a large exposure time, 840 h, against 700 h for cyclic tests. The same failure mechanisms were observed in both cases. However, the reduced number of cooling steps in the quasi-isothermal oxidation tests slowed down the mass loss. Considering that the cooling step is the main factor for the oxide layer spallation, the quasi-isothermal test is closer the cyclic oxidation than the isothermal test. Their analysis justifies the HH steel mass loss under quasi-isothermal tests in the present study, but at a lower rate than that observed for cyclic oxidation tests. Nicholls and Bennett [11] summarized some cyclic oxidation tests methodologies. According to the definition mentioned by them,

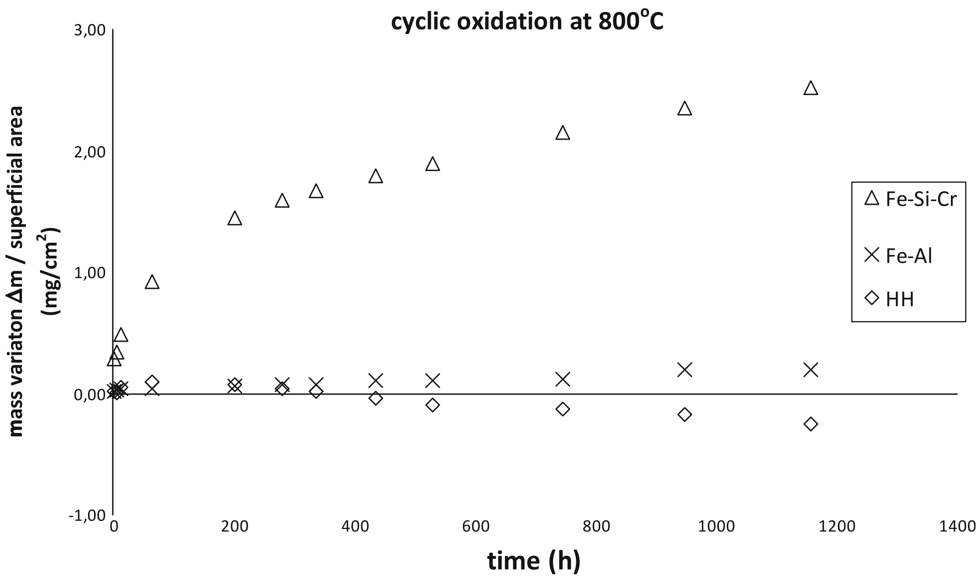


Fig. 2 Mass variation under cyclic oxidation at 800 °C, comparing Fe–Si, Fe–Al, and HH alloys

the quasi-isothermal test is not a cyclic test only because it does not have an exact frequency, since the period in the cycles can be as long as 8,000 h. Thus, for quasi-isothermal test they used the term "discontinuous oxidation", which refers to tests where the samples are not hold inside the furnace and are cold for few and short periods of time comparing with cyclic tests.

Wear tests

Figures 3 and 4 presents the average mass loss of samples tested in the dry sand/rubber wheel equipment. Figure 3 compares the three materials tested after oxidation during 700 h at 800 °C. Under such condition, the Fe–Si suffered less wear (near 8 mm^3), overcoming the HH steel (~9.5 mm^3) and the Fe–Al alloy (~12 mm^3). The Fe–Si behavior shall be due to Cr presence because Cr increases the wear resistance by forming carbides. The less resistance presented by Fe–Al, comparing with stainless steel, had already been observed by Maupin et al. [9], who compared test results of Fe–Al intermetallic under pin-on-drum with stainless steel results (AISI 304) from the literature, obtaining a higher wear rate for the intermetallic alloy. On the other hand, Johnson et al. verified higher resistance of Fe_3Al than that of AISI 304 under sand/rubber wheel tests, suggesting that these materials have the same level of wear resistance.

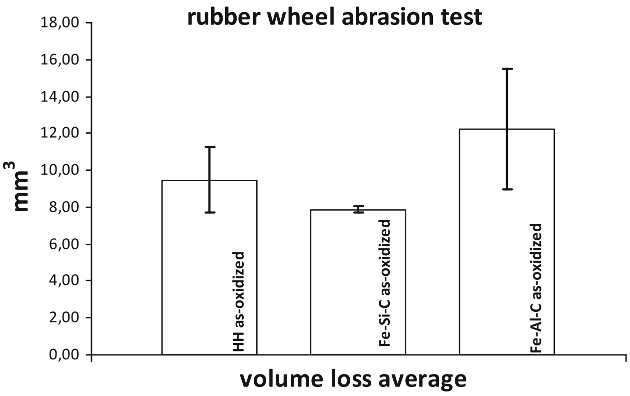


Fig. 3 Volume loss under rubber wheel abrasive wear test: comparative to the three materials after oxidation

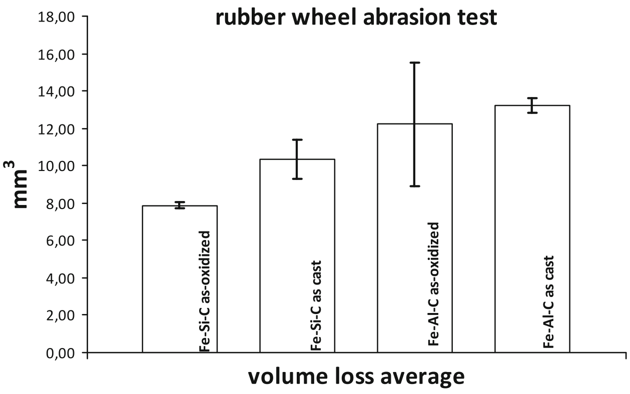


Fig. 4 Volume loss under rubber wheel abrasive wear test: comparative between as-cast and after oxidation specimens

Figure 4 compares the materials Fe–Si and Fe–Al under two conditions: as-cast and as-oxidized. On the as-cast condition the tendency was the same as previously observed, with better results for Fe–Si. The as-cast samples suffered more wear than oxidized samples, due to the higher wear resistance of the oxide layer, probably because the higher hardness as cited in the literature [10].

The higher resistance of oxidized specimens is evident for Fe–Si alloy. However, for Fe–Al, due to the scattered results, we cannot affirm that the wear resistance is lower than that of the as-cast material. Another important factor to be considered is the layer thickness. Considering a finite oxidized layer material, just the initial wear is influenced by such layer. For Fe–Al the layer is quite thin what explains the lower wear resistance of the material. Xia et al. [10] evaluated the mass loss under pin-on-disk test for a Fe–Al alloy and observed that alumina layer protects the sample against wear attack. To prove their hypothesis oxidized samples were tested for different periods of time, and thus with different alumina layer thickness. They found wear resistance improvement as the layer thickness increased. The improvement was also observed in other wear forms such as, abrasive wear, adhesive wear, and severe oxidative wear. In several situations the wear and oxidation occur at the same time, thus the wear resistance improvement due to the oxide layer can be quite interesting. If the oxidation rate is superior to wear rate there will always be an oxide layer to improve the wear resistance.

Conclusions

The intermetallic alloys, Fe–Al and Fe–Si, presented superior results than HH steel under quasi-isothermal and cyclic oxidation tests. The best result was for the Fe–Al alloy.

It was observed that quasi-isothermal oxidation tests generate almost the same damage than cyclic oxidation tests. The quasi-isothermal test produced longer sample's life because the lower number of cooling cycles than those applied for cyclic oxidation tests.

Under wear abrasive tests the Fe–Si alloy obtained the better result, while the Fe–Al alloy did not achieve the same resistance of HH steel.

The as-oxidized samples had better results in the wear tests than the as-cast samples. The Fe–Si alloy presented the best results mainly because the thicker oxide layer.

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