Wear Resistance Improvement of Copper Alloys Using a Thermochemically Obtained Zinc-Rich Coating

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Abstract—It has been developed a thermochemical process applied on copper alloys: brass and bronze, using pure zinc powder, obtaining a zinc-rich wear protective coating. The layers obtained by a diffusion process, on brass (alloy C36000) and bronze specimens (alloy SAE 62), were characterized using a scanning electron microscope, EDAX microanalysis, Vickers microhardness, X-Ray diffraction analysis, and sliding wear test. The chemical analysis showed a layer composition of 62 % Zn and 38 % Cu, on average. The microhardness for thermochemical treated brass was 496HV and 598HV for bronze; thus, a microhardness increase for brass is 468% and 532% for bronze. It was made an X-Ray diffraction analysis, confirming the results obtained with the chemical analysis and crystalline structure for coating. It showed the presence of Cu$_3$Zn$_8$ and Cu$_6$Zn$_5$ phases. The wear tests demonstrated that treated specimens show better wear resistance than non-protected specimens.

Index Terms—Copper Alloys, Thermochemical Treatment, Wear Resistance, Zinc-Rich Coating.

I. INTRODUCTION

Methods used to protect a surface against surface deterioration processes involve the need of a coating that isolates the material against damaging situations. There are metallic coatings that can isolate the metal from the atmosphere, creating a barrier layer, which offers higher corrosion resistance. Zn, like other anodic metals, protects the substrate metal even if there are imperfections in the coatings, a situation that does not occur with the cathodic coatings. There are different methods to obtain a zinc rich coating on a surface: sherardizing [1,2], thermal spray, electrogalvanizing, zinc-rich paints [3], hot-dip galvanizing [4].

Copper base alloys, such as bronzes, have been used for many years in applications where they can work under wear conditions. If this behavior is improved, the service life of such parts can be extended. Brass has limited wear resistance and it is also an alloy of engineering interest. A better behavior against wear is expected to increase applying the thermochemical treatment proposed in this work, extending the useful life of these materials.

The hot-dip galvanizing of steels is a well-known process. There is an essential available content in the literature about properties, phases, and microstructure in these coatings. The presence of Zn-Fe intermetallic compounds in different zones of the coatings has been found, as a product of diffusion processes occurring during hot-dip galvanizing.

The literature about diffusion processes using pure zinc powder on steels, which is commercially known as sherardizing, is fewer. In their research, N. Pistofidis [4-6] et al., studied the diffusion mechanism of Zn in Fe, using sherardizing, as an alternative to the hot-dip galvanizing method. The cementation method, performed at 400 °C, provides uniform coating composed mainly of two layers, achieving corrosion resistance similar to that obtained by the hot immersion method. These zones of the layer are gamma ($\gamma$-Fe$_{11}$Zn$_4$) and delta ($\delta$-Fe$_7$Zn$_3$), corresponding to those indicated in the Fe-Zn Phase Diagram. It was observed, through thermal analysis techniques, that the process is carried out in three stages when ammonium chloride is used as the activator. In the first stage (carried out at 193.9 °C), which is endothermic, the transformation of $\alpha$-NH$_4$Cl to $\beta$-NH$_4$Cl is carried out, and the NH$_4$Cl decomposes into NH$_3$ and HCl. During the second stage (which is exothermic, developed at 248.6 °C), Zn$^{2+}$ ions are formed - mainly - as a ZnCl$_2$. Finally, in the third stage (which occurs at 264.1 °C), the Zn is deposited by an endothermic reaction on the ferrous substrate through the decomposition of ZnCl$_2$. It is also pointed out that the corrosion resistance found in sherardized samples is similar to that found in hot-dip galvanized parts and that the corrosion mechanism in the case of hot-dip galvanizing is associated with a pitting mechanism, and in the case of sherardizing, with phenomena of stress corrosion.

Natrup and Graf extensively describe the sherardizing process [7], pointing out the appropriate temperature conditions, the preparation procedures for the surfaces to be coated, and the characteristics of the zinc powders to be used. They also describe in detail the phenomena of diffusion that can occur during sherardizing, the presence of the gamma ($\gamma$) and delta ($\delta$) phases, and the high hardness that can be reached in these coatings is highlighted; in a first hypothesis, it is attributed to the possible presence of a significant number of defects in the coating. An additional

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aspect - to take into account - [8] - the importance of appropriate heat treatment selection - to sherardized steel parts - to ensure no impact in mechanical properties; it is also necessary - the study of the effect of - using different halides on the growth of sherardized layers [9].

In the ‘40s, Ernest Kirkendall studied the phenomena of diffusion in the Cu-Zn pair, and subsequently, other works [10-14] have confirmed the basic ideas that emerged from that study. The investigations described that Zn diffuses faster in the Cu than the opposite case, by a difference of several orders of magnitude.

Recent works [15-17] have focused on determining Cu and Zn diffusion coefficients. The evaluation of the diffusion mechanisms in grain boundaries in the Cu-Zn system and through grains has been considered one of the most viable routes. X. L. Kong et al. [18] used a powder compacting process to create a Cu-Zn alloy nanocrystalline layer on a copper substrate. Non-lubricated wear tests were carried out, and evaluations were performed using scanning electron microscopy (SEM) and X-Ray diffraction. It was found that by performing the process at 300 °C, the layer exhibited excellent mechanical and tribological properties due to the high microhardness achieved [19-21]. It was found, utilizing X-ray diffraction, that a nanocrystalline ZnO film is formed, which reduces the coefficient of friction with stable and durable layer properties.

II. MATERIALS AND METHODS

A. Coating fabrication

Two stainless steel AISI 304 containers - were used for the experiment. Each container will lodge six specimens. The containers were covered with zinc powder (99.5% purity) and NH₄Cl as an activator (5 and 10%). Subsequently, the pieces were covered entirely, and another layer was added so that containers were wholly encircled by zinc powder and activator. The diffusion process temperature had a notorious effect on the created coating. The zinc powder did not melt quickly, so that, each microscopic particle of zinc undoubtedly melted, but the particles did not join each other to form a melt of any considerable size; this phenomenon is attributed to the influence of a thin layer of oxide surrounding each dust particle [5,7].

The experiments in the oven were carried out at a temperature of 400 °C for 8 hours in a Lindberg electrical oven (50/60 Hertz, 240 Volts, 5900 Watts, temperature max. =1500 °C). The specimens designated for wear test were treated in groups of 6 specimens per container. Likewise, were treated specimens with different geometrical forms. The final coating was observed using a scanning electron microscope (SEM) Phillips XL20.

B. Metallographic analysis

Thermochemically treated specimens were cut crosswise to obtain samples and perform a metallographic analysis using the scanning electron microscope (SEM) Phillips XL20 [12]. The specimens were then assembled in bakelite and prepared using silicon carbide sandpaper with different sizes (180, 200, 360, 400 600 and 1000), then polished with alumina (3.0 μm and 0.5 μm). Samples were evaluated in the scanning electron microscope to obtain topography, morphology, and thickness of the coating.

C. Chemical Analysis

Using the EDAX microanalysis - probe (the equipment has the capacity to detect chemical elements, starting from sodium, atomic number 11), - coupled to the Philips XL 20 SEM, chemical analysis was carried out on the thermochemically-treated specimens in different areas of the coating and the base metal, along a cross line from the end of the protective layer to the original material, which determined the elemental chemical composition (weight). Measurements are made at kV: 25.00 Tilt:15.00 Take-off:47.56 Tc:20 Detector Type: SUTW-Sapphire Resolution :158.51 Lsec :300

D. Microhardness

Vickers microhardness tests were performed using the Leitz Wetzlar Germany 8068 equipment, on the coating of brass and bronze specimens, with - ten indentations in the base metal. It was used a load of 100 g during 10 s.

E. X-rays analysis

For the X-ray diffraction evaluation, the Bruker D8-Advance Powder Diffractometer was used, using Cu K radiation, graphite monochromator, with the Diffplus B_S software, using Powder Method. Likewise, measurements were done using a Siemens 5000 diffractometer, operating at 35 kV and 25 mA with a crystalline graphite monochromator, using a copper lamp. Samples were scanned at a rate of 2° per minute between 20 and 100°.

F. Sliding Wear test

Adhesive wear tests were performed using the Cygnus II Equipment Model 51 with 2000 rpm, 220V, and 60 Hz, designed for dry or lubricated wear testing and test loads from 0.5 to 5 kg that can be applied in axial form. In this case, loads ranging from 0.1 kg to 0.5 kg were used. The tests were carried out by placing and evaluating 3 pieces simultaneously, according to the configuration of the equipment. Speeds from 180 rpm to 300 rpm were varied in the tests, which gives the distance traveled and is used to determine the wear of it. The contact disc is AISI D2 heat-treated tool steel. The test radius is 0.035 m. To determine the weight of the test pieces, and Ohaus electronic scale balance with a resolution of 0.0001 g was used every 60 s.

III. RESULTS

A. SEM, Thickness and Chemical analysis

Fig. 1 shows a treated and an untreated sample after the pin on disc test. Some aspects are highlighted. There is a change in surface hue, which is associated with the presence of a zinc-rich layer. It is also observed the difference in the surface condition of the worn brass part with evidence of plastic deformation, and the absence of plastic deformation in the treated sample.
The thickness on the different coating layer for the brass specimens had an average of 80 μm; this can be observed in Fig. 2:

![Brass samples with and without coating. The edge is observed in the upper part of the uncoated specimen, a burr is generated after pinning on disc test, by plastic deformation.](image)

In the case of bronze, it was observed that the thickness of the coating layer is more significant than the obtained for brass, with an average of 142 μm; this can be observed in Fig. 3. For the case of the white zones, the results of the analysis are very similar, which can be interpreted as evidence of the possible presence (in small quantity) of phase β' of the Cu-Zn system.

![Coating layer on brass obtained by zinc diffusion, 8 hours at 400 °C.](image)

![Coating layer on bronze obtained by zinc diffusion, 8 hours at 400 °C.](image)

Fig. 4, 5, 6, and 7 show the chemical composition for the coatings. Tables I, II, III and IV show the semi-quantitative results of the respective EDAX analyzes.

**TABLE I: WEIGHT PERCENTAGE FOR COATING IN BRASS (FIG. 4)**

<table>
<thead>
<tr>
<th>Element</th>
<th>Wt %</th>
<th>At %</th>
</tr>
</thead>
<tbody>
<tr>
<td>CuK</td>
<td>38.13</td>
<td>38.8</td>
</tr>
<tr>
<td>ZnK</td>
<td>61.87</td>
<td>61.2</td>
</tr>
</tbody>
</table>

![Results of the chemical analysis obtained with the EDAX equipment in the zinc-rich coating layer on brass.](image)

**TABLE II: WEIGHT PERCENTAGE FOR COATING IN BRASS (“WHITE” AREAS) (FIG. 5)**

<table>
<thead>
<tr>
<th>Element</th>
<th>Wt %</th>
<th>At %</th>
</tr>
</thead>
<tbody>
<tr>
<td>CuK</td>
<td>52.1%</td>
<td>52.1%</td>
</tr>
<tr>
<td>ZnK</td>
<td>47.9%</td>
<td>47.19%</td>
</tr>
</tbody>
</table>

![Results of the typical chemical analysis obtained with the EDAX equipment in “white” areas of the zinc-rich coating layer on brass.](image)

**TABLE III: WEIGHT PERCENTAGE FOR COATING IN BRONZE (FIG. 6)**

<table>
<thead>
<tr>
<th>Element</th>
<th>Wt %</th>
<th>At %</th>
</tr>
</thead>
<tbody>
<tr>
<td>CuK</td>
<td></td>
<td></td>
</tr>
<tr>
<td>ZnK</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
CuK  37.09  37.76  
ZnK  62.91  62.24  

<table>
<thead>
<tr>
<th>Element</th>
<th>Wt %</th>
<th>At %</th>
</tr>
</thead>
<tbody>
<tr>
<td>CuK</td>
<td>50.86</td>
<td>51.57</td>
</tr>
<tr>
<td>ZnK</td>
<td>49.14</td>
<td>48.43</td>
</tr>
</tbody>
</table>

Fig. 7. Results of the typical chemical analysis obtained with the EDAX equipment in "white" areas of the zinc-rich coating layer on bronze.

**TABLE IV: WEIGHT PERCENTAGE FOR COATING IN BRONZE ("WHITE" AREAS) (FIG. 7)**

With all the obtained information, the Activation Energy Q and Diffusion Coefficient D$_0$ were calculated. For brass:

$$Q = 0.00422234 \left( \frac{\text{kJ}}{\text{kg mol}} \right)$$  \hspace{1cm} (1)

$$D_0 = 1.70358 \times 10^{-13} \left( \frac{\text{m}^2}{\text{s}} \right)$$  \hspace{1cm} (2)

In the case of bronze, the Activation Energy Q and Diffusion Coefficient D$_0$ calculated were:

$$Q = 0.576164 \left( \frac{\text{kJ}}{\text{kg mol}} \right)$$  \hspace{1cm} (3)

$$D_0 = 6.35177 \times 10^{-13} \left( \frac{\text{m}^2}{\text{s}} \right)$$  \hspace{1cm} (4)

As can be seen, the coating layer on thermochemical treated bronze (average of 142 μm) is thicker than thermochemical treated brass (average of 80 μm).

**B. Microhardness**

Using a load of 100 g, the Vickers microhardness was measured for each specimen; for the coating on brass an average value of 496±165 HV was obtained, in this case the base material had a hardness of 106±4.4 HV. It is possible to obtain a linear correlation between the two variables with an R$^2=0.41$. For bronze, the coating microhardness was around 598±65 HV, with a linear relationship of R$^2=0.69$ and 113±6.4 HV for the base metal. Fig. 8a shows the microhardness results for the coating on brass and Fig. 8b on bronze.

The above results clearly indicate the positive effect of the incorporation of the Zinc rich coating on the bronze, since the average microhardness increased 5 times. In addition, the HV measurements also reached higher values at larger distances of penetration, which is in accordance with the data of activation energy calculated for this system. The increment of microhardness is attributed to the formation of the γ phase composition (63%) within the Cu-Zn system. Therefore, the diffusion process induces the enrichment of this phase mainly on brass alloy.

**C. X-rays analysis**

Results obtained by the X-Ray analysis, are presented in Fig. 9 and 10. It is confirmed an existing crystalline structure in the coating. The presence of phases of the Zn-Cu system and of various oxides of Cu and Zn, including the coexistence of the CuO and Cu$_2$O structures were identified.
D. Sliding wear test

The results (Fig. 11) show a substantial increase in the wear resistance achieved in the treated specimens compared to those without treatment (higher for bronzes). For bronzes, when velocities of 180 rpm and 0.1 kg load were applied, the weight loss varied between 0.39 and 6.26 times higher in untreated specimens. With the increasing speed at 300 rpm and a load of 0.5 kg, the order of magnitude for weight loss reaches 18.58 times more in untreated specimens compared to untreated samples. For brass, at low speeds, weight loss is about 2.75 times higher in untreated specimens, but when tested at 300 rpm and a load of 0.5 kg, the weight loss is 76.6 to 164.4 higher in the untreated parts compared to the treated. The percentage of weight loss for brass after the tests is within 24% for pieces without treatment, and 0.15% for the treated. For bronze, the percentage of weight loss is higher than 8% for the untreated samples compared to 0.45% for those of bronze that were subjected.

IV. DISCUSSION

The coating layer is more prominent in bronze (average of 142 μm) than in brasses (average of 80 μm). The crystalline structure for Cu is fcc (face-centered cubic), for Zn is hcp (hexagonal close-packed), and for Sn is bct (body-centered tetragonal), and the atomic packing factor (APF) for Cu and Zn is 0.74 and for Sn is 0.64. Then, there is more probability of finding free spaces in the crystal structure of bronze (Cu-Sn) than in brass (Cu-Zn) for atoms diffusion. For brass specimens, it was obtained composition on the coating of 38.13% Cu and 61.87% Zn weight percentage. For the bronze case, it was obtained a result of 37% Cu and 63% Zn weight percentage. Thus, the coating layer is uniform and very similar in composition comparing brass and bronze (see Fig. 4 to 7).

Considering the Vickers microhardness analysis for brass and its coating, the increase in microhardness is within 468% (Fig. 8). The results were taken along the coating and base metal, from 20 μm (coating) to 500 μm (base metal). The length of the coating for brass is 80 μm average and the
microhardness decrease after that magnitude. Then, the obtained coating and the increase in microhardness are correlated between them as reported elsewhere [22]. For the bronze case, the increase in microhardness is in the order of 532%. The data were obtained along with the coating and base metal, from 20 µm (coating) to 400 µm (base metal). In the case of bronze specimens, the thickness of the -coating was 142 µm average.

The results of the Vickers microhardness test are in agreement with this asseveration. The hardness in α phase is attributed to the local distortion, and in β phase, to the difference in the type of lattice. Hardness in the γ phase is attributed to the small atomic volume and the loss of symmetry. This loss of symmetry, coupled with the fact that in γ brass, is obtained the maximum amount of distortion, will probably account for the exceptional hardness of γ brass as compared with the hardness of the other alloys in the series. As discussed in [22-31], the complex crystalline structure of this phase is associated with a high defect density that would also contribute to greater coating hardness. Both coated materials show a linear relationship between their microhardness and the distance from the surface, in the case of bronze there is a correlation index of 0.41, for brass a value of 0.69 is obtained. These values provide a clear estimate of the relationship between the model and the response variable (microhardness).

The X-Ray analysis (Fig. 9 and 10), shows a cubic structure of Copper Zinc (Cu66Zn36, JCPDS card No 50-1333) phase with Miller’s index equal to a=b=c=3.69612 Å. The results report the presence of a Cu2Zn2 -like structure, phase (JCPDS 65-6566, crystal system cubic, space group I43m, cell parameter 8.85-8.89 Å). These results agree with literature [22-31], and show the presence of the γ phase of the Zn-Cu system, which is a harder phase.

In the case of bronze, the X-Ray analysis show – the formation of a cubic structure of Copper Zinc (Cu66Zn36, JCPDS card No 50-1333) with Miller’s index equal to a=b=c=3.69612 Å and a phase Cu2Zn8 (JCPDS 65-6566, crystal system cubic, space group I43m, cell parameter 8.85-8.89 Å). It is – confirmed - for brass case and according to various references [22-31], the presence of γ phase of the Cu-Zn system. Also, a cubic structure of Copper Oxide in which a=b=c= 4.26960 Å is perceived. The identification of phases in corresponding diffraction patterns pointed out the presence of γ phase in the coating, which confirms data found by the chemical analysis from the microprobe. Additionally, the appearance of a ZnO phase is evidenced according to the ICSD 01-079-0208 reference standard, located at the peak 2θ=31.49° and in a preferential direction (100), which coexists with Cu66Zn36, Cu3Zn8 and CuO in the peak 2θ=72.22° with preferential direction (004), this oxide has a hexagonal crystalline structure with space group P63mc. In turn, it is possible to detect CuO according to the ICSD 01-080-0076 reference standard in the peak 2θ=72.22° with Miller index (311), this oxide has a space group number 15 (C2 / c), and structure monoclinic crystalline. Despite the composition, both alloys show the presence of Cu oxides (and ZnO), this feature is associated with the oxophilic behavior of copper atoms that allows higher oxygen adsorption [32].

In the other hand, the wear behavior of brass without a zinc-rich layer shows evidence of plastic deformation, and bronze without zinc-rich layer does not show deformations. A detailed interpretation of the wear mechanisms is beyond the scope of this work. Thus, in a following study, the wear mechanisms and the role, especially of Cu and Zn oxides as suggested by Kong et. al., [19], will be evaluated with the use of microscopic techniques.

It has been confirmed – with SEM/EDAX and X-ray diffraction - the formation of a zinc-rich γ phase, - with high hardness properties. It should be noted that, in sliding wear test, at low speeds with low loading (0.1 and 0.15 kg), the coating did not disappear, only to 500 rpm with 0.5 kg of axial load the coating was removed. The microhardness increment can be related to the improvement of wear resistance (Fig. 11 and 12).

V. CONCLUSIONS

The thermochemical treatment applied on copper alloys in this research, using pure powder zinc (~ during 8 hours - at 400°C), obtained a coating layer of 80 µm average for brass and 142 µm for bronze.

The coating layer is uniform on the entire surface of the specimen, regardless the geometry of this one. The chemical analysis determined a chemical composition on the coating layer of 62 % Zn and 38 % Cu average for both brass and bronze. Using the data obtained in the chemical analysis and the values for Atomic Packing Factor (APF) for Cu (fcc structure with APF=0,74), Zn (hcp structure with APF=0,74) and Sn (bcc structure with APF=0,64 approximately), it can be inferred that there is more free space for the atoms for diffusion in bronze than in brass; this is the reason of bigger coating thickness - in bronze. The X-Ray analysis confirmed the phases, and also determined that exist crystalline structures in the coating: Cu66Zn36, Cu3Zn8 phases, and copper oxide in small quantity.

The increase in microhardness for brass is in the order of 468% up to 532%. The composition of the Zn (63% weight) in the coating corresponds to the γ phase of the Cu-Zn phase diagram and confirms - the reason to explain the significant increase in microhardness. This, in turn, confirms the results of the wear tests, which indicate that the weight loss experienced by an untreated brass sample, is much higher than that experienced for the case of thermochemically treated parts. Similar behavior was observed in the case of bronze. It can be concluded that the process developed in this work is a technology that improves the wear resistance of these copper alloys properly and, in this sense, allows them to extend their useful life and increase their possibilities of application.

REFERENCES


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She carried out a postdoctoral research at the CENISA group of the National University of Mexico from 2012-2014 developing hybrid sol-gel coatings doped with nancontainers and corrosion inhibitors loaded in nanofibers produced by the electrospinning technique. Her current position since 2015 is at the CENISA group of the Engineering School of the National University of Mexico.
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